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# Corrosion

*devoted entirely to*

## CORROSION

### *Research and Control*

PUBLISHED MONTHLY AS ITS OFFICIAL JOURNAL, BY THE NATIONAL ASSOCIATION OF CORROSION ENGINEERS, INC., AT HOUSTON, TEXAS, U. S. A., TO PROVIDE A PERMANENT RECORD OF PROGRESS IN THE CONTROL OF CORROSION AS DESCRIBED IN PAPERS PREPARED FOR THE ASSOCIATION AND FROM OTHER SOURCES.

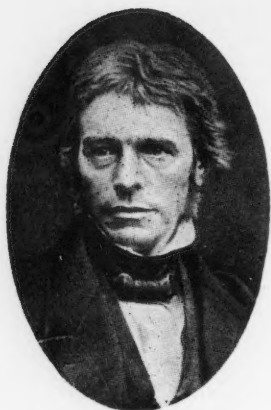


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## THIS MONTH'S COVER

• Inside of a 42-inch OD steel pipe after receiving a life-lengthening corrosion-resistant lining. The internal coating is applied while the pipe is rotated at a rate of 760 linear feet per minute (approximately 70 rpm). Lined pipe is but one of several corrosion-prevention measures utilized in such as the water, sewage, chemical, oil, gas and paper and pulp industries to avoid costly repairs and replacements with their usually attendant plant shutdown, or interrupted customer service. Photograph courtesy Mayes Brothers, Houston, Texas.



*Michael Faraday, F.R.S.\**

## *Michael Faraday and the “box in the storeroom”*

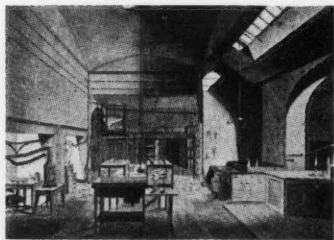
IN THE YEAR 1822 Professor Michael Faraday of the Royal Institution finished up some work on alloys he had made in the laboratory, and put the remaining specimens away in a wooden box. He labelled the box with his own hand and sent it to the storeroom.

Faraday then went on with his famous researches on electricity and magnetism, and in the course of time he completely forgot about that old box. Many years later, in March 1863, the distinguished metallurgist Dr. John Percy inquired about the specimens. But Faraday, who was then 72 years of age, answered . . .

“I wish I could have the further pleasure of helping you, but I have forgotten all about the paper on alloys, and cannot at all call to mind where any of the specimens are. I rather think they . . . must be lost by this time, for I do not know how to trace them.”\*

### *A pioneer student of corrosion-resistant alloys*

The box was thus forgotten; and it remained forgotten for over a century. In 1930 it was re-discovered, and the 79 specimens in it were carefully studied by Sir Robert Hadfield. With the help of entries in Faraday's diary, it became clear that the great scientist had been systematically investigating the effect of certain alloying elements in conferring corrosion resistance and other desirable properties on alloys. Faraday was evidently the first to study the nickel-iron



*Faraday's Laboratory at the Royal Institution.\**

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# Non-Destructive Methods For Determining Metal Plate Thickness\*

By J. G. Kerley\*

THIS article was prepared with the feeling that corrosion engineers may use the instruments described to minimize loss of operating time normally required for inspection purposes. In a few cases it may be possible to make all necessary measurements while a unit is in operation, and avoid shut-down until loss of metal necessitates parts replacement. In a still larger number of cases it may be possible to appreciably reduce shut-down time by increasing the speed of inspection. These are possibilities for saving money, and are attractive to industry<sup>1</sup>. However, they require a judicious choice of instruments because *there is no all-purpose instrument* for the many inspection problems found in industry today. Most instruments have definite fields of application and definite limitations, and these are usually due to the physical principles employed.

Thickness gages have been built that utilize the following physical

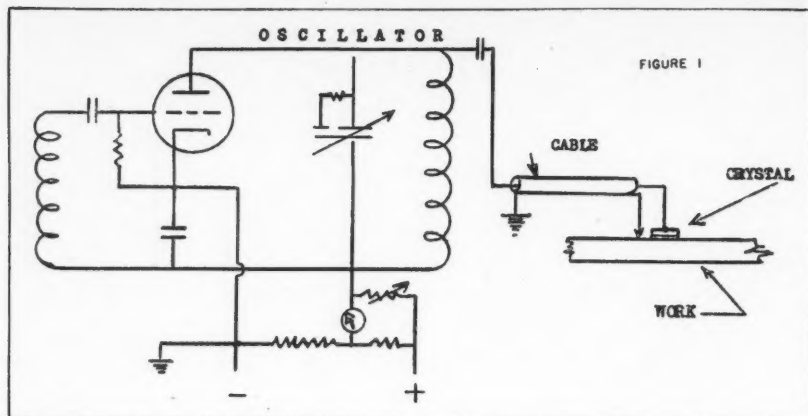
phenomena. Ultrasonic vibration, thermal conductivity, magnetism, electrical resistivity, electrical induction, and penetrating radiation. It is readily apparent that such a broad field cannot be discussed in great detail in the space available. It is proposed to accept the peculiarities of these principles as facts, and to discuss instead the methods in which they are used. This best can be done by describing an instrument in each field, and even this must be pared down to a brief description of the operating principle and method, coupled with a few remarks on advantages and limitations.

## Supersonic Vibration Methods

During recent years instruments have been perfected both here<sup>2, 3, 5, 6</sup> and abroad<sup>4</sup> that utilize supersonic (more appropriately, ultrasonic) vibration as a basic principle. Methods employed are based on two facts: first, that sound travels through metals at characteristic rates which are not appreciably influenced by ordinary variations in temperature, alloy content, etc.; and second, that a sound wave in metal is reflected back to its source by barrier surfaces like echoes in the at-

\* A paper presented at the Annual Meeting of NACE in Chicago, Ill., April 7-10, 1947. Original paper compiled in collaboration with B. G. Crane, formerly of Ethyl Corporation.

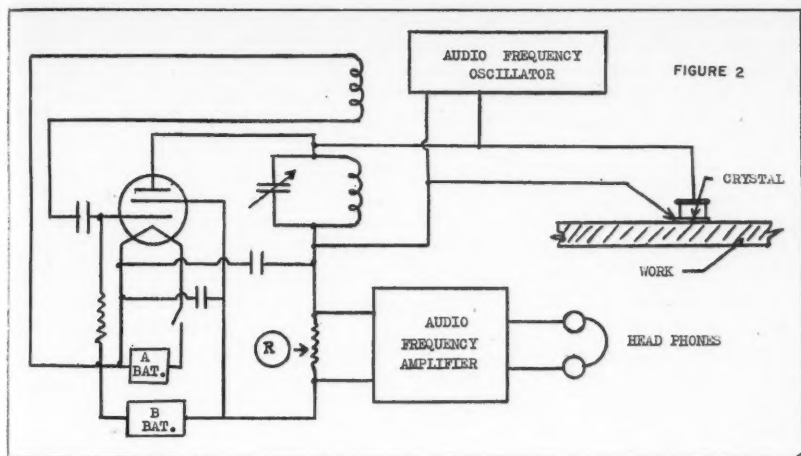
\* Senior Engineer and Chairman of the Measurement and Control Committee, Shell Oil Company, Inc., New York, N. Y.



mosphere. Since the velocity of sound in all metals is known or can be determined, thickness can be measured by determining the time necessary for a pulse of sound to make a "round-trip" from one surface to the other and back to the origin.

It is not always necessary to measure the time directly. One instrument than can be used for measuring thickness of very thin plate eliminates time measurement by determining the fundamental frequency at which the plate vibrates in resonance. This is accomplished by equipment illustrated in Figure 1. The variable-frequency electronic oscillator shown at the left, generates alternating voltage at frequencies governed by the position of the tuning-capacitor knob on its calibrated scale. This voltage is impressed across the faces of a small X-cut quartz crystal, which converts the electrical energy into mechanical vibrations and projects them into the sample with which it is held in contact.

In operation, a thin film of grease or oil is maintained between the crystal and sample to assure good sound transmission. The oscillator is then tuned with the knob until the sample vibrates in resonance. A unique method is employed in determining this resonant condition. Motional impedance of the crystal is such that as long as the sound waves in the sample are "out of phase," so to speak, with the vibrating crystal, the power required to drive the crystal is very small. However, when the frequency is such that a reflected sound wave returns to the origin just in time to support vibration of the crystal (this, of course, is a resonant condition) the power to drive the crystal is maximum. This results in a very abrupt, sharp peak in the power-output curve for the oscillator. Therefore, a milliammeter as shown in the oscillator circuit indicates a resonant point at its peak reading. A frequency reading taken at such a peak will be either the fundamental resonant frequency or one of its harmonics.



The difference in frequency between any two successive harmonics is always equal to the fundamental. Therefore, thickness can be determined by taking frequency readings at two successive points of maximum current, subtracting the lower from the higher, and entering the difference in the equation:

$$T = \frac{S}{2F}$$

where  $T$  = the thickness of the sample in inches,

$S$  = the velocity of sound in the material, in inches per second, and

$F$  = the fundamental resonant frequency in cycles per second.

This technique was developed by General Motors Corporation<sup>2</sup> for inspecting hollow metal airplane propellers. Their instrument, called the "Sonigage," is a special-purpose device, and is not available as a general inspection tool. There is, how-

ever, a quite similar instrument available for inspection services.

This unit is known as the "Audiage," and is marketed by Branson Instruments, Inc., Danbury, Conn. A schematic diagram is shown as Figure 2. It is apparent that the basic principles and components are the same as those of the "Sonigage," except that the indicating milliammeter has been replaced by an audio frequency modulating circuit and a set of head phones that result in an audible signal at the resonance point. Like the maximum reading on the "Sonigage" indicator, this signal is quite sharp at resonance, so that with a little training, an operator can take readings rapidly and accurately.

The tuning dial of the "Audiage" is calibrated to read in megacycles; and, in addition, a thickness scale in inches of steel is plotted opposite corresponding fundamental frequency values so that when the fundamental has been determined the

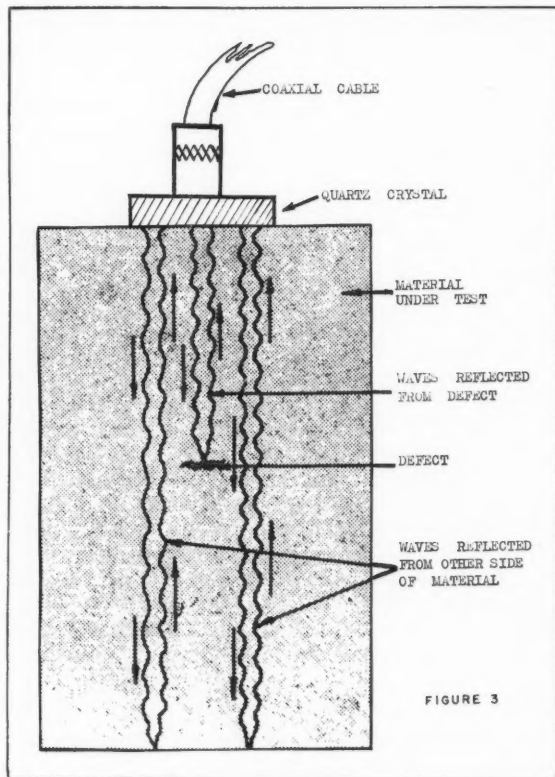
thickness can be read directly. For materials other than steel, appropriate factors are used as multipliers. The unit is portable, battery-powered, and is operated by one man.

With both of these instruments some surface preparation is necessary. Readings cannot be obtained where the opposite surface is severely pitted. Scale or other material on the opposite side does not affect the accuracy.

Resonance instruments are used only for thickness measurement, usually in relatively thin plates. There is on the market today an instrument that measures time directly, and combines thickness measurement with detecting flaws in heavy castings and shapes. Manufactured and marketed by Sperry Products, Inc., Hoboken, New Jersey, this device is known as the "Reflectoscope."<sup>5,6,7</sup> As can be seen from Figure 3, the name is indicative of the method employed. It might appropriately be called a "sonic radar."

If there are no flaws in a sample, a pulse of sound projected downward as shown will be reflected at the opposite face and will return

to the origin. If it is then picked up electrically and projected on a properly marked, or calibrated, cathode-ray oscilloscope the thickness of the sample can be read directly from the oscilloscope screen. Moreover, if there is a subsurface fault in the sample beneath the crystal, wave energy will be reflected back from it to the origin, and will appear as an additional "pip" of light on the oscilloscope, whose distance from zero will be proportional to the depth of the flaw. Furthermore, the size of the flaw can be approximated from the character of the "pip."



When the "Reflectoscope" is used in this fashion, continuous wave energy is not employed. Energy from the oscillator is beamed into the sample in pulses of a few microseconds duration, with the crystal acting as a transmitter. The rate of these "bursts" is 60 per second. The rest of the time the crystal acts as a receiver for returning pulses, converting them into electrical energy and projecting them on the oscilloscope screen.

#### Accurate to Depths of 10 Feet

Used in this fashion, the instrument is best suited to flaw detection in such heavy pieces as railroad car axles, and has been extensively used for detecting fatigue cracks in such equipment. It is very accurate up to depths of 10 feet, and has been used up to 20 feet.

By using continuous wave energy and tuning the oscillator, the resonance method described earlier can be used. In this case, resonance of the sample is indicated by a standing wave of "pips" on the screen; thickness being indicated by the distance between wave crests.

Equipment utilizing similar methods has been used in other countries<sup>4</sup> for thickness measurement, flaw detection in welded seams, and other similar forms of inspection.

#### Thermal Conductivity Methods

In general, all thermal conductivity methods employ the same basic principle: i.e., the application of heat at a controlled rate to the material under inspection for definite periods of time, and measurement of the temperature rise in the sample. The rise in temperature is a function of the specific heat, ther-

mal conductivity and the volume of the sample. Since the first two can be assumed constant for a given material and the area of application is usually fixed, temperature readings are indicative of thickness.

#### Principles of Operation

Because the writer is unaware of a commercially available instrument using this method, suffice to describe one of the most prominent developments in this field. This method, known as "Thermoflux," was developed by A. V. deForest of Massachusetts Institute of Technology<sup>5</sup> in collaboration with Fidelity and Casualty Company of New York. Heat was applied by electrical induction through two legs of a magnetic yoke held in contact with the sample. The a-c energizing coil was wound around the body of the yoke, and a temperature sensitive element — usually a thermocouple — was affixed to the sample between the legs.

Heat was induced in the sample for definite periods of time, dependent on the approximate sample thickness. A period of 30 seconds was found satisfactory for thickness ranges from 0.25 to 0.75 inch, while 120 seconds were required for thicknesses from 0.75 to 2 inches.

#### Several Sources of Error

Obviously this instrument was subject to several sources of error. In the first place, since heating was accomplished inductively, the magnetic reluctance of the sample influenced the heating effect of the field. For ordinary steels this was found to be of negligible importance. What was more serious was the inability to fit the yoke faces snugly against

the surface. This also affected the heating since it varied the reluctance of the magnetic circuit. Some difficulty was also encountered in affixing the temperature-sensitive element to the sample. Moreover, it was found that measurements near a free edge were subject to error, probably due to distortion of the magnetic path.

Unfortunately, development of the instrument was never completed, due to the war. Several models were built which showed considerable promise. During the war the method was turned over to the U. S. Navy, who recently released it. Magnaflux Corporation, who took over this activity, have announced no new developments.

### Magnetic Methods

Measurement of the magnetic characteristics of steel plates is apparently rather popular with designers of thickness gages. During the past decade several types of magnetic measurements have been employed, both for thickness determination and for detection of flaws.<sup>9</sup> Such thickness gages range from one which measures the force required to pull a magnet of known strength free from the surface of a metal plate, to one employing a rather ingenious "magnetic bridge" circuit. In the former, the force required is assumed to be a function of the plate thickness. In the latter, readings are obtained directly by measuring the amount of steel core that must be inserted in an electromagnetic "balancing" coil, to exactly balance the magnetic flux from a similar electromagnetic "measuring" coil held in contact with the sample.

The difficulties encountered in these methods are quite similar to those discussed in "Thermoflux" above, namely variations in reluctance of the magnetic path due first to the alloy content, stresses, etc., in the metal itself; second, to variations in the air gap due to improper fitting pole pieces; and finally, to path distortion from shape variations, free edges, etc., in the sample. These difficulties coupled with the fact that saturation is hard to achieve in thicker pieces have limited thickness measurements to five-eighths of an inch and less.

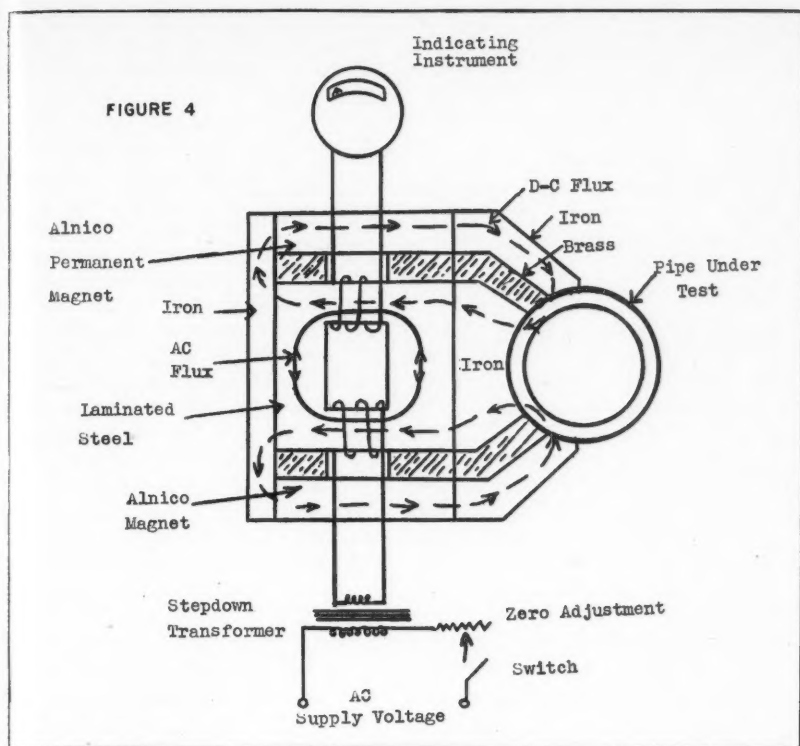
### Magnetic Principle

Despite these difficulties, very successful instruments have been built utilizing magnetic measurements. One of these, General Electric Company's Type MS-A Gage, designed specifically for measuring the wall thickness of 1- to 2-inch pipe, is illustrated in Figure 4. The unit consists of a gage head, to which a heavy saddle carrying the contacting pole pieces is attached, and an indicating instrument calibrated in inches thickness.

### Resembles Saturable Reactor

In operating principle, the unit closely resembles the well-known saturable reactor. Flux from a powerful Alnico magnet passes through the saddle and close-fitting pole-pieces into the sample of pipe, saturating it. To complete the magnetic circuit, it then passes through the core of the gage head, about which is wound a special a-c transformer, the primary of which is powered from a 110-volt 60-cycle source, and the secondary is tied to the indicator. Since the uni-direc-





tional flux from the magnet tends to block the a-c flux and its resultant transformer action, the amount of flux necessary to saturate the sample is determined immediately by the meter reading which, since saturation is achieved, is a direct indication of sample thickness.

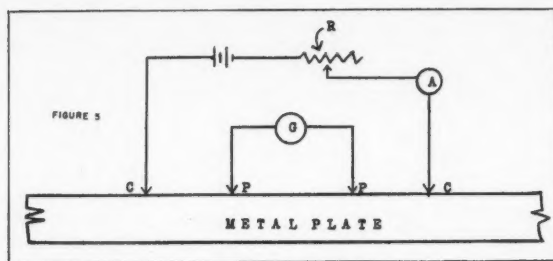
Doubtless, much of the success of this device is due to its snug-fitting pole pieces, which minimize reluctance errors due to air-gap variations. A separate saddle is used for each pipe size. Saddles can be quickly changed since they are attached by two screws. Although it has been built only for 1-to 2-inch

pipe sizes, the unit can be built to meet requirements.

Magnetic instruments enjoy several advantages over others. They can be made very rugged and portable. Readings are not influenced by materials on either surface if the magnetic path is not affected.

### Electrical Resistivity Methods

Perhaps the oldest and most widely used basis for thickness gages in the simple method<sup>10,11,17</sup> of measuring the resistance encountered when direct current is passed through a section of conducting sample. The basic principle is illustrated



in Figure 5. If a flow of direct current is established in the circuit C-R-A-C, potential drops will be set up in all parts of the circuit due to resistance to current flow. The potential drop in the metal plate C-C will be directly proportional to the resistance of the material and the length of the electrical path, and inversely proportional to the cross-sectional area encountered by the current. Obviously then, potential points P-P could be tied into this circuit just above C-C, and from the voltage drop read on G, a determination of plate thickness could be made. It can be readily seen, however, that the resistance of contacts C-C would be a possible source of large errors. Moreover, the distribution of the flowing current in the metal would not be uniform.

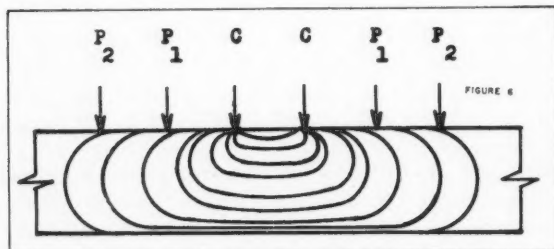
For this reason, in all the instruments of today, separate potential contacts are provided at points of

uniform current distribution. Most manufacturers provide contact heads with fixed spacing where the distance between contacts is determined by the range of the instrument—or in other words, by the thickness to be

measured. Current is usually supplied from a 6- or 12-volt storage battery, and potential readings are made on a microammeter. Although the procedure may be reversed, it is customary to maintain a fixed current flow and use variations in potential as indications of thickness. Generally the instrument is not calibrated directly, but potential readings are entered on curves that have been plotted from actual readings on calibrated samples. Quite often, this introduces an error that is unavoidable where the 4-point method (2 current contacts and 2 potential contacts) is used.

Due to composition, alloy content, etc., the unit resistance of the various samples inspected will seldom exactly equal that of the samples used in plotting calibration curves. Although the resulting error is usually small, it should be eliminated, if possible, and the principle illustrated in Figure 6 affords a method of doing so.

Usually, in a single steel plate, the resistance is uniform throughout the entire plate. If, in such a plate and at the same current value, poten-



tial readings are obtained at two fixed points such as  $P_1P_1$  and  $P_2P_2$ , a ratio of these readings eliminates resistance by cancellation, since its value is the same for both voltage readings. Furthermore, such a ratio provides a very nice curve for thickness measurement as can be seen from Figure 7, where the ratio  $P_1/P_2$  is used as ordinates and thickness is shown as abscissae. Note the effect of contact spacing on the range of the instrument.

This is commonly referred to as the "six-contact" method or "two-potential" method, and a variation of this technique is used in "Electroflux," an instrument manufactured and marketed by R. C. Scott, Belmont, Mass. In the Scott instrument, provision is made in the head for making a two-potential reading. From this reading the effect of resistivity can be determined and a multiplier calculated. The four-point method is then utilized, since it is faster, and readings are corrected by application of the factor.

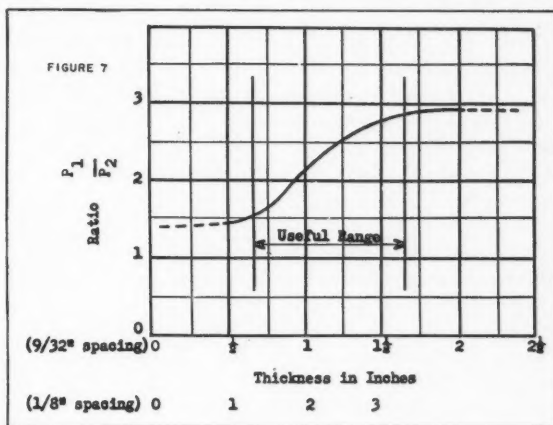
"Electroflux" is a modern instrument incorporating the latest developments to minimize thermal effects and other inherent errors. It is powered by a 6-volt airplane storage battery, and a stable electronic amplifier is used to increase sensitivity and minimize errors in potential readings. Five contact heads are provided, giving the instrument a range of from 0.125 to 2.25 inches. Con-

tacts are arranged at corners of a square, and the spacing is usually approximately equal to the thickness of the plate under inspection.

With instruments utilizing this principle, metal thickness only is obtained, and readings are not affected by scale or back-up liquid. The exterior surface should be cleaned, however, and extreme care should be taken to make good contacts at the head.

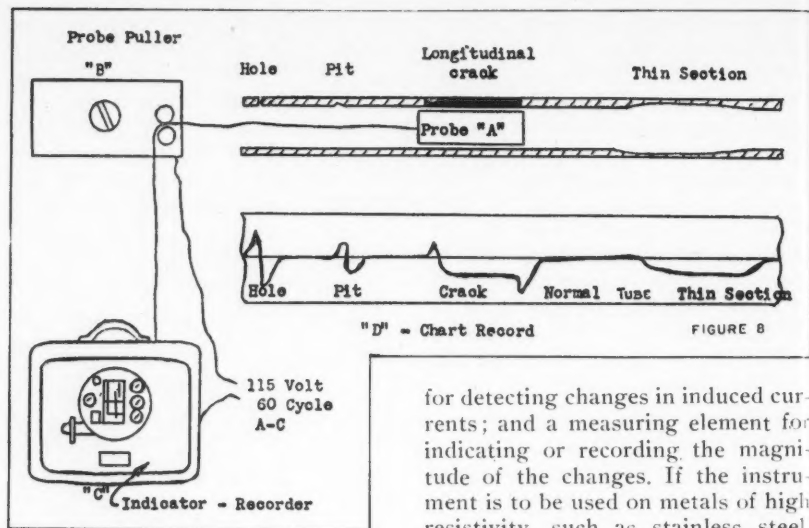
### Electrical Induction Methods

The general principle employed in



electrical induction methods of testing has been known for years, and has been used as the basis for a wide variety of instruments designed for specific problems.<sup>13,14,15,16</sup> It is briefly described as follows:

If a conductor lies in an alternating magnetic field, there will be induced in that conductor electrical eddy currents the magnitude and pattern of which are dependent on three things: 1), the characteristics of the magnetic field; 2), the electrical conductivity of the con-



ductor, and 3), the physical characteristics, such as dimensions of the conductor. If a magnetic field of known constant frequency, intensity and pattern is applied to a conductor, the physical character of the conductor can be determined by measuring the magnitude and pattern of the resulting eddy currents. Because the electrical conductivity of a homogeneous material is fairly constant, variations in these eddy currents are reliable indications of structural flaws, such as change in granular structure, pits, cracks and variations in dimensions.

Construction details of instruments utilizing this principle vary widely, design of a particular instrument usually being that most suitable for the task involved. All such devices are similar, however, in that each incorporates the three necessary components—an electromagnetic exciting source: a "pick-up"

for detecting changes in induced currents; and a measuring element for indicating or recording the magnitude of the changes. If the instrument is to be used on metals of high resistivity, such as stainless steel, usually some form of electronic amplification is inserted between the current pick-up and the final indicating means.

Of the many devices using this principle, few have been designed as field inspection tools. One of the most outstanding devices of this type is that designed, built, and marketed by Shell Development Company, Inc., and called the "Probolog."

### Special Purpose Unit

This instrument was designed for a specific purpose, the non-destructive testing of the tubes incorporated in the "bundle" of a heat-exchanger or condenser without removing the tubes from the "bundle," and in many cases, without removing the "bundle" from the shell. This is accomplished by making the test from the inside bore of the tube. Figure 8 shows the three elements that make up this device, and their arrangement in general test work.

Both the energizing source and "pick-up" are incorporated in the probe "A" shown inside a typical tube. This probe is pulled along the tube at a uniform rate by an electrically-driven probe puller "B." The probe is connected by cable to the electronically-operated, null-balanced, bridge-type indicator-recorder "C," where the strip-chart record "D" is obtained. Since the rate of the probe puller is synchronized with chart travel, this record is a profile of the tube, showing metal removed. The amount of metal removed at any particular spot is related to the deviation of the pen from normal; and the length of the damaged section, as well as its location along the length of the tube can be determined from the record by simple measurement.

#### Wide Sensitivity Range

Because it may be desirable to record only those tubes having flaws above a certain magnitude, the instrument is equipped with a neon-indicating light having a wide sensitivity adjustment. This light may be set to flash to faults of a certain size. The probe may then be pulled through the various tubes until a tube is encountered having flaws exceeding the desired minimum. A record is then made of the tubes causing the light to flash, to determine further the extent of the flaws and the general condition of the tube.

The instrument is equipped with a stepped, wide range sensitivity adjustment to meet the needs of various materials. Materials for which the instrument is recommended include practically all metallic conductors except the ferro-magnetic group. Although some measure-

ments are possible at present on steel tubes with the instrument, its use as a general inspection tool for this service has not been perfected, and the manufacturer does not recommend it for this purpose.

This apparatus is particularly useful in detecting such insidious forms of corrosion as dezincification and other microscopic flaws. It will detect flaws that are apparent only after considerable magnification under a microscope. Some practice and experience with the unit are necessary for an intelligent interpretation of chart records.

#### Penetrating Radiation Methods

When a beam of penetrating radiation, such as X-rays or gamma rays, is passed through a section of material, it is broken up into two types of wave energy. Part of the primary rays will pass directly through the section, and the amount passing through will depend on the initial penetrating power of the beam as well as the density of the material and the thickness of the sample. The rest of the beam will be deflected or scattered, and its penetrating power decreased by the action of the electrons that make up the atoms of the material. If it is not totally absorbed by the atoms, this energy will emerge from the surfaces as radiation having considerably lower penetrating power than the primary beam. Part of this deflected radiation will emerge from the same side of the sample that the primary beam entered, and this portion is known as back-scattered radiation.

#### New Device Uses Gamma Radiation as Primary Beam

Industrial radiologists have for a number of years utilized measure-

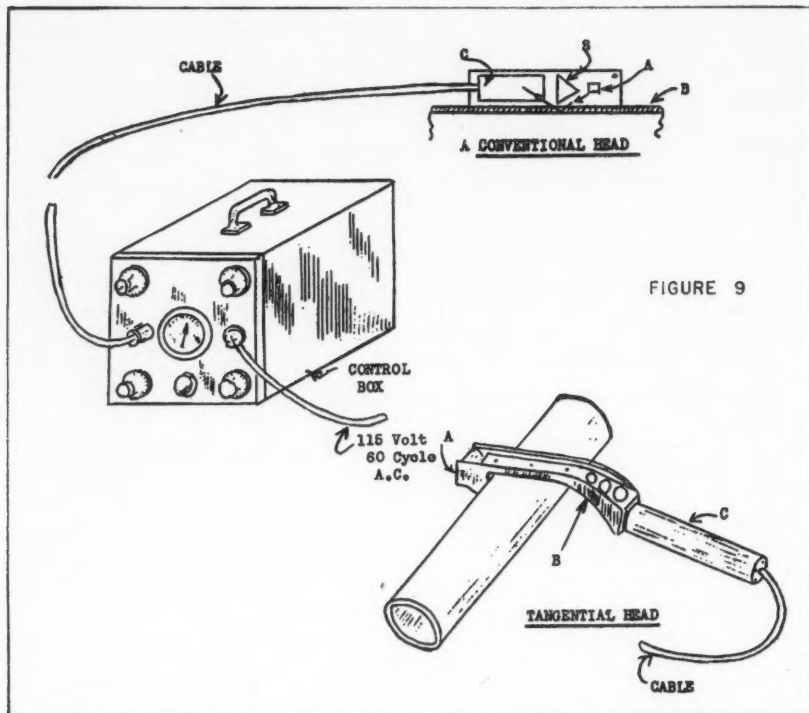


FIGURE 9

ment of the residual beam—that is, ray absorption—in the form of X-ray and gamma-ray photography, which is quite common throughout industry.<sup>17,18</sup> Only in recent years, however, has the principle of radiation measurement been incorporated in simplified instruments suitable for field use. The “Penetron,” a device developed by The Texas Company and marketed by Engineering Laboratories, Inc., Tulsa, Okla., which utilizes gamma radiation as a primary beam, is an excellent example of ray-sensitive thickness gages, since one of its detector heads measures back-scattered radiation and the other measures direct absorption.<sup>19,20</sup>

The principle of operation of this unit may be seen in Figure 9. The conventional head, which measures back-scattered radiation, utilizes one milligram of a commercially available radium salt located at the apex of a conical shield designated “S” in the diagram. This source, “A,” emits a beam of radiation, designated “B,” which passes into the sample, with the resultant effects described previously. The resulting back-scattered radiation is picked up by a sensitive gamma-ray detector at “G” and causes the detector to release pulses of electricity proportional to the rate at which the gamma rays are received. These pulses are amplified in



TABLE I  
Characteristics of Available Thickness Gages

NAME	MANUFACTURER	PORTABILITY (WEIGHT)	POWER REQUIREMENTS	AVERAGE AREA OVER WHICH READINGS ARE TAKEN	RANGE OF THICKNESS	% ERROR	REMARKS
Audifuge	Benson Ints., Inc. Joe's Hill Road Danbury, Mass.	Portable. Total Wt. - 14 lbs.	Battery operated	Area of crystal; varies up to 1 sq. in.	0.125" to 12"	15% actual thickness	Can be operated by one man. Surface should be prepared. Not affected by scale, liquids or solids on opposite side.
Reflectoscope	Sperry Products Inc. 15 & Willow Ave. Hoboken, N.J.	Mounted on cas- sets Wt. - 175 lbs. complete	115 volt 60 cycle AC. (2 amps reqd.)	Area of crystal	$\frac{3}{8}$ " to 10" (Laminations down to 1/8" by resonance method)	Depends on Operator - $\frac{1}{16}$ " in depth up to 10 ft.	Used principally on heavy billets and shapes. Very good for fatigue crack testing.
Type MS-A	General Electric Co. Schenectady, N.Y.	Portable. Approx. 10 lbs.	115 volt 60 cycle AC.	Area under pole pieces; varies	0 to 1/4"	$\frac{1}{16}$ " in $\frac{1}{4}$ " in	Limited to 1" to 2" pipe; special purpose instru- ment, but can be built to suit.
Electroflux	R. C. Scott 340 Common St. Belmont, Mass.	Portable; 3 pieces; total weight 37 lbs.	Battery operated	Area under con- tacts; varies from 1/64 sq. in. to 4 sq. in.	1/8" - 2 1/4"	15% actual thickness	5 contact heads supplied for variety of thick- nesses. Not in- fluenced by ma- terial on opposite side.
Probolox	Shell Development Co. Inc. 90 W. 50th St. New York, N.Y.	Portable; 2 cases; total weight 100 lbs.	115 volt 60 cycle AC.	Cylindrical tube section of very short length de- pending sensi- tivity setting.	Depends on material being inspected, sensitivity setting, etc.	Not quanti- tative	Special purpose instrument - for non-ferrous tubes only.
Ponatron	Engineering Labora- tories, Inc. 705 Kennedy Bldg. Tulsa, Oklahoma	Portable. Total weight, approx. 15 lbs. (35 lbs. plus cable and heads)	115 volt 60 cycle AC.	1 sq. in.	Standard head 3/4" tangential head 2"	15% actual thickness	Affected by de- posits on opposite side. Requires 25 seconds for a read- ing.

a small pre-amplifier circuit built into the head, and are then transmitted over the connecting cable, usually 75 feet long, to the control station. In the control box, the pulses are again amplified, rectified to direct current, and are measured by a microammeter, which serves as a final indicating means.

The reading on the microammeter dial is directly related to the density and thickness of the sample. Since density is involved, the instrument is not calibrated directly in thickness, but is used with curves for various materials that have been plotted from samples of known thickness. Along with density, surface curvature influences the shape of these curves; for instance, a curve for 4-inch pipe will be somewhat different than one for steel plate of equal thickness.

Several different types of clamps can be supplied for holding the conventional head in place, and a pneumatic clamp with extension handles is available for use in internal inspection of pipe and tubing.

### Conclusion

It has not been possible to discuss all of the instruments available for inspection services. The writer knows of several, and there are doubtless others with which he is not familiar, deserving of detailed discussion. However, to include them would require a much more comprehensive and lengthy article. It may, however, serve the engineer as a guide to the general principles and characteristics of available instruments, and enable him to select the device best suited to his needs. He can then contact the manufacturer for more details.

When used with the tangential head, the same control box uses direct radiation absorption in thickness measurement; also illustrated in Figure 9. The radiation beam "B" passes directly through the sample to the detector at "C" and the pulses are transmitted to the control box as described above.

Curves similar to those described earlier are plotted for various materials and shapes, and thickness data are obtained from the curves in the same manner as for the conventional head. Curves for the two heads are not interchangeable.

In using the "Penetron" for general inspection purposes, it is important to remember that this instrument actually measures mass, and serious errors can result from foreign deposits, such as process scale, on samples being measured. Likewise, if not compensated for, liquids in pipes or tubes being measured can cause error. Furthermore, since radiation is used, a certain minimum length of time—about 25 seconds—is necessary for reliable readings.

Pertinent information on the devices discussed has been tabulated in Table I. It is hoped that this tabulation includes most of the important details that were necessarily deleted from the body of the paper. Only those instruments available at present are included.

In conclusion one salient fact is pointed out. The information in Table I should serve as supporting evidence for a statement made in the introduction, and reiterated here: "There is no all-purpose instrument for the many inspection problems found in industry today."

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Editor's note: This paper including trade names of instruments is presented to give the reader a general idea of the scope of some relatively new tools. As pointed out by the author, one instrument in each field has been selected and discussed.

# Ferric Ion Corrosion During Acid Cleaning\*

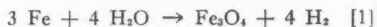
By F. N. Alquist\*, J. L. Wasco\* and H. A. Robinson\*

THE USE OF ACID solvents to remove scale, sludge, corrosion products and other accumulations from heat-exchange units has increased tremendously in popularity during the last ten years. The contributing factors toward this trend are: 1) the ability to clean all parts of a unit without dismantling; 2) savings in outage and manpower; 3) existence of companies who offer complete acidizing service, and 4) the research sponsored by the service companies to uncover new corrosion inhibitors, methods of operation and solvents for deposits which are difficult to remove.

In the course of research along this line, a type of corrosion was uncovered which is inherent to the acid cleaning of equipment that contains ferric oxide deposits. This corrosion is "ferric ion corrosion"—corrosion of the parent metal by ferric ions from the iron oxides as they reduced to ferrous ions.<sup>1,2</sup>

Most steel heat-exchange equipment using water or steam becomes fouled with iron oxides on the water side of the unit. Magnetic iron oxide,  $\text{Fe}_3\text{O}_4$ , is a corrosion product in high

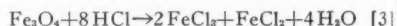
pressure boilers. It is formed by the water dissociation reaction:



Red iron oxide  $\text{Fe}_2\text{O}_3$ , is formed in any boiler or exchange unit which uses water that has not been completely deaerated:

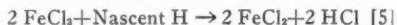


During the acid cleaning of a heat-exchange unit, these iron oxides are dissolved, forming ferric ion salts:

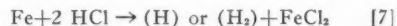


However, the drain acid solutions give no test for ferric ion; all of the iron present is in the ferrous state.

There are two reactions which reduce the ferric ion to ferrous:



The hydrogen in nascent state is produced by reaction of iron with acid:



Laboratory tests have proved that these reactions take place simultaneously during acid cleaning. Reaction [5] was shown to occur by corrosion tests in which hydrogen

\* A paper presented at the Annual Meeting of NACE in Chicago, Ill., April 7-10, 1947.  
\* The Dow Chemical Co., Midland, Mich.

**TABLE I**  
**Fe<sub>2</sub>O<sub>3</sub> Removed from Utility Boilers by Acid Cleaning**

BOILER	Surface Area Sq. Ft.	Number of Acid Cleanings	Average Lbs. of Fe <sub>2</sub> O <sub>3</sub> Dissolved per Cleaning
A.....	20,000	2	500
B.....	20,000	3	410
C.....	42,070	2	720
D.....	42,070	1	918
E.....	12,000	1	390
F.....	12,000	1	280
G.....	12,000	1	310
H.....	20,000	1	1196
L.....	20,000	1	265

evolution measurements were made. The amount of hydrogen produced by the reaction of inhibited acid on steel is materially reduced when a ferric salt or ferric oxide is added to the acid. Tests have shown that as much as a 50 percent decrease in hydrogen evolution can be effected by the presence of a ferric salt.

Equation [6] is shown to be true by the increase in corrosion of steel in inhibited acid when ferric ions are present. From reactions [5], [6], and [7] it is obvious that the economic loss is metallic iron.

The acid corrosion inhibitor which protects the metal from attack by acid has no restraining effect on the corrosion of the metal by the ferric ions. The extent of the ferric ion corrosion is dependent entirely on the amount of ferric iron oxides dissolved. (See Table I). The present methods of acid cleaning are controlled so that the sum of the acid

corrosion and the ferric ion corrosion is not excessive.

A search was begun for some method of reducing or eliminating the ferric ion corrosion so that acid cleaning would be even safer. A number of ways were immediately suggested:

1) Placing a large bundle of scrap steel shavings in the top drum of the boiler being acid cleaned. The added amount of metal exposed to the acid solution would favor reduction of ferric ions by the scrap metal instead of the metal of the unit. This method was actually put to a test in acid cleaning a boiler, but analysis of the solutions showed no reduction in the amount of corrosion of the equipment.

2) Circulating the acid solvent through a cartridge filled with a substance capable of reducing ferric ions to ferrous. The suggested reducing agents were iron or tin filings.

3) Adding some compound directly to the acid, which would reduce or combine with the ferric ion immediately it was formed and before it attacked the parent metal. This method was the most promising due to its advantage of dispersing the reducing agent throughout the entire unit.

Specifications were established and the ferric ion reducing agents were

**TABLE II**  
**Corrosion Rates in lbs./sq. ft./day of Mild Steel in inhibited HCl Solutions for 16 Hours**

Grams Glyoxal	Grams Fe <sub>2</sub> O <sub>3</sub>	2.5 HCl			5% HCl			10% HCl		
		135° F.	150° F.	175° F.	135° F.	150° F.	175° F.	135° F.	150° F.	175° F.
0.0	0.0	.007	.008	.030	.010	.014	.025	.018	.024	.052
0.0	1.5	.010	.012	.040	.021	.027	.031	.025	.033	.059
0.6	1.5	.009	.011	.035	.017	.027	.023	.021	.025	.050
1.2	1.5	.008	.008	.029	.016	.022	.020	.021	.024	.049

TABLE III

Glyoxal as Ferric Iron Reducing Agent with Various Acid Corrosion Inhibitors. Tests at 170° F. in 10% HCl on Mild Steel.

INHIBITOR		LBS./SQ.FT./DAY		
		0.0 g. Fe <sub>2</sub> O <sub>3</sub>	2.4 g. Fe <sub>2</sub> O <sub>3</sub>	2.4 g. Fe <sub>2</sub> O <sub>3</sub>
0.4 %	Type	Glyoxal	Glyoxal	Glyoxal
A	.....	.085	.107	.090
B	.....	.056	.087	.068
C	Aldehyde.....	.244	.243	.167
D	Aldehyde.....	.069	.082	.069
E	Aldehyde.....	.042	.055	.044
F	Nitrogen base.....	.048	.079	.062
G	Nitrogen base.....	.029	.053	.043
H	Aldehyde-amine condensation product.....	.104	.089	.097
I	Sulfoxide.....	.016	.052	.044
J	Mercaptan.....	.017	.090	.055
K	Nitrogen base.....	.044	.077	.044
L	Nitrogen base.....	.060	.086	.055
M	Aldehyde.....	.070	.081	.070
N	Aldehyde + nitrogen base.....	.055	.121	.097
O	Aldehyde + nitrogen base.....	.045	.080	.053
P	Nitrogen base.....	.045	.062	.045

rated on their ability to meet them as follows:

1) Ability to largely decrease or eliminate ferric ion corrosion in acid cleaning heat-exchange equipment.

2) High solubility in hydrochloric acid solutions.

3) Compatibility with hydrochloric acid, metal, acid corrosion inhibitors and all other agents used in acid cleaning.

4) Non-toxic, in itself or its by-products.

5) Freedom from formation of any objectionable tars, gases, odors or solids during acid cleaning.

6) Ease in handling—preferably a liquid having lower freezing point than water and fairly low vapor pressure.

7) Economically feasible.

Glyoxal, a dialdehyde, (CHO)<sub>2</sub>, was found to be the most desirable of all the compounds tested. It is a good reducing agent, soluble in

TABLE IV

Organic Materials Tested for Ability to Reduce Ferric Iron in 15% HCl.

4.0 grams Fe<sub>2</sub>O<sub>3</sub> added to 300 g. 15% HCl at 150° F. with stirring. Twice the theoretical amount of organic reducing agent was added. Oxidation potential measured with platinum-calomel cell just before addition of organic material and after 30 minutes.

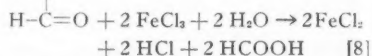
Compound	E <sub>1</sub>	E <sub>2</sub>
Amidol.....	+0.470	+0.450
Catechol.....	.470	.405
Glyoxal.....	.470	.430
Hydroquinone.....	.470	.420
Phenyl hydrazine.....	.470	.290
Pyrogallol.....	.470	.270
Aniline.....	.470	.475
Benzaldehyde.....	.470	.468
Butadiene sulfone.....	.470	.455
Chloral hydrate.....	.470	.470
Citric acid.....	.470	.470
Crotonaldehyde.....	.470	.470
Formaldehyde.....	.470	.465
Furfural.....	.470	.465
Glucose.....	.465	.465
p-Hydroxyazobenzene.....	.465	.465
Hydroxylamine.....	.470	.470
Lactic acid.....	.470	.470
Naphtheneic acid.....	.470	.470
8-Naphthol.....	.465	.455
Oleic acid.....	.472	.470
Phenol.....	.470	.470
Phloroglucinol.....	.470	.470
Resorcinol.....	.470	.470
Tannin.....	.470	.460
Tartaric acid.....	.470	.470
Triphenylchloromethane.....	.470	.470

E<sub>1</sub> = Initial oxidation potential in volts.  
E<sub>2</sub> = Final oxidation potential in volts.

hydrochloric acid solutions, easy to handle, not toxic, and it forms no objectionable by-products.

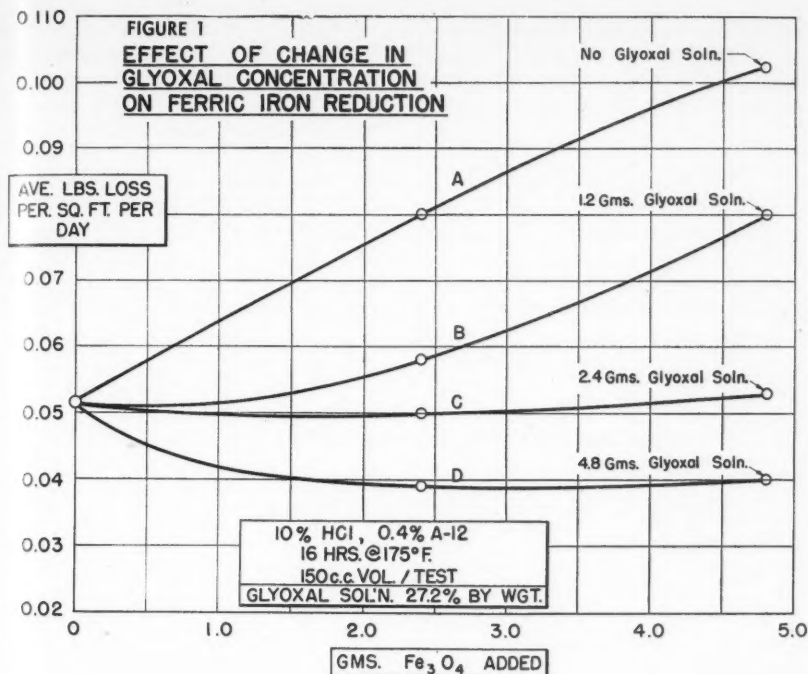
Glyoxal is available commercially in an approximately 30 percent water solution. This solution is yellow, slightly viscous, and has a specific gravity of 1.2.

The main reaction of glyoxal with ferric chloride appears to be as follows:



Ferric chloride is reduced to ferrous chloride, while glyoxal is oxidized to formic acid. All of these compounds are soluble in the acid solutions and in no way interfere with the normal acidizing procedure.





### Method of Testing

The laboratory testing technique was based on the ability of a compound to decrease the corrosion of steel test specimens in inhibited hydrochloric acid solutions containing iron oxide.

The corrosion tests were made in duplicate in eight-ounce, wide-mouth glass jars. One hundred fifty milliliters of a hydrochloric acid solution of an arbitrary concentration and 0.6 cc. or 0.6 g. of an acid corrosion inhibitor were placed in the jar; the jar was covered with a watch glass and placed in a constant temperature bath. The temperature of the acid solution was allowed to come to equilibrium in the temperature bath. Then reagent grade  $Fe_2O_3$  or

$Fe_3O_4$  and the ferric ion reducing agent were stirred into the acid. Mild steel test strips were added immediately and the tests allowed to remain undisturbed for 16 hours.

The test specimens were prepared from 1-inch by  $\frac{1}{8}$ -inch by 16-foot S.A. 1010 steel stock, which was sawed into pieces  $2\frac{3}{4}$  inches by 1-inch by  $\frac{1}{8}$ -inch. These pieces were stamped with number dies for identification, sand blasted, and stored in a desiccator. Test specimens were weighed to one milligram before and after testing.

After the test, the specimens were rinsed in water, scrubbed clean with scouring powder and a dental plate brush, rinsed in water and acetone, dried and re-weighed.

The corrosion rate was calculated in pounds of iron lost per square foot per day from the following equation:

$$\frac{3.2 \text{ (grams lost)}}{(\text{Original weight of strip in grams})} = \text{lbs. lost/sq. ft./day}$$

This equation was based upon the premise that since the width and thickness of all strips were alike, the surface area of the specimens was directly proportional to the original weight of the strips within the accuracy of the test.

### Laboratory Data

Figure 1 illustrates the effect of  $\text{Fe}_3\text{O}_4$  and glyoxal on the corrosion rates of steel in inhibited hydrochloric acid. The corrosion rate was increased considerably by the addition of  $\text{Fe}_3\text{O}_4$ , but this ferric ion corrosion was entirely eliminated by the subsequent addition of glyoxal. The corrosion rate of the inhibited acid plus  $\text{Fe}_3\text{O}_4$  can be reduced below that of inhibited acid alone by the addition of glyoxal.

Glyoxal is effective over a wide range of temperature and concentrations of hydrochloric acid. Table II shows data at 135 to 175° F., and at hydrochloric acid concentrations

TABLE V  
Aldehydes as Reducing Agents

Corrosion Tests in 10 Percent Inhibited Hydrochloric Acid on Mild Steel for 16 hours at 175° F.

g. $\text{Fe}_3\text{O}_4$	g. Aldehyde	Corrosion Rate Lbs./Sq. Ft./Day	Tar Formation
0.0	0.0	.044	None
1.5	0.0	.055	None
1.5	2.4 g. glyoxal	.039	None
1.5	4.0 g. formaldehyde	.042	None
1.5	4.0 g. acetaldehyde	.037	Some
1.5	4.0 g. crotonaldehyde	.023	Heavy
1.5	4.0 g. benzaldehyde	.061	Some
1.5	4.0 g. furfuraldehyde	.039	Very Hvy.
1.5	4.0 g. phenylacetaldehyde	.038	Some
2.4	1.5 g. chloral	.084	None
2.4	2.0 g. chloral hydrate	.083	None

of 2.5 to 10 percent, which are the limits of acid cleaning conditions for a unit containing a normal iron oxide deposit. Laboratory tests have shown glyoxal to be effective even in 25 percent hydrochloric acid. The corrosion rates (Table II) changed very little at lower temperatures when  $\text{Fe}_3\text{O}_4$  or glyoxal was added. This was due to the limited solubility of  $\text{Fe}_3\text{O}_4$  at 135° F. in hydrochloric acid solutions.

Glyoxal can be used with most commercial acid corrosion inhibitors (Table III). It is more efficient with some than others, but in all cases it did reduce some of the ferric ion corrosion. Glyoxal, however, was useless when tested in 10 per-

TABLE VI  
Other Reducing Agents

Reducing Agent	INHIBITOR X			INHIBITOR Y		
	0.0 g. $\text{Fe}_3\text{O}_4$	2.4 g. $\text{Fe}_3\text{O}_4$	4.8 g. $\text{Fe}_3\text{O}_4$	0.0 g. $\text{Fe}_3\text{O}_4$	2.4 g. $\text{Fe}_3\text{O}_4$	4.8 g. $\text{Fe}_3\text{O}_4$
None	.044	.077	.088	.044	.076	.112
1.2 g. $\text{SnCl}_2$	....	.064	.069	....	.053	.070
3.2 g. $\text{TiCl}_3$	....	.067	.069	....	.086	.112
0.75 g. $\text{NH}_4\text{CNS}$	....	.059	.075	....	.058	.091
1.0 g. $\text{NaH}_2\text{PO}_2$	....	.060	.060	....	.055	.074
1.2 g. Catechol	....	.070	.066	....	.063	.085
1.0 g. Hydrazine sulfate	....	....	....	....	.075	.086

Corrosion Rates in lbs./sq. ft./day at 175° F.

cent concentrations of sulfuric, nitric and phosphoric acid solutions.

After the above laboratory work was completed, glyoxal was added to inhibited hydrochloric acid during the acidizing of a three-drum, bent-tube boiler. This treatment consisted of pumping 15,500 gallons of 7 percent inhibited hydrochloric acid at 165° F. into the boiler. Fifty gallons of 32 percent glyoxal solution was proportioned into the ingoing acid. The solution was allowed to remain in the boiler for eight hours before it was drained. Samples of the drain indicated that 55 percent of the glyoxal was oxidized by the ferric ions. This amounts to the saving of 83.2 pounds of iron to the boiler being cleaned.

#### Data on Other Compounds

Before glyoxal was tried, considerable data had been accumulated on various other compounds. Table IV lists a group of organic compounds which were tested for their ferric ion reducing properties by measuring the oxidation reduction potential. Pyrogallol and phenyl hydrazine are the two compounds of this group which showed the greatest promise. Pyrogallol was later shown to be an erratic reducing agent by corrosion tests, while phenyl hydrazine proved to be very good and has been used successfully in acid cleaning a boiler. Phenyl hydrazine has the disadvantages of high toxicity and cost.

Phenyl hydrazine is extremely toxic and would be hazardous to use, although the phenyl hydrazine hydrochloride which is formed immediately upon its addition to hydrochloric acid is considerably less toxic. In the above-mentioned boiler

treatment, 260 pounds of phenyl hydrazine were added to 10,000 gallons of inhibited 7 percent acid. Fifty percent of the phenyl hydrazine was oxidized to benzene and nitrogen, equivalent to a saving of 135 pounds of iron to the boiler.

Aldehydes, as a general class of organic compounds, have been known as reducing agents for a long time. Upon investigating the more commonly known compounds of this group (Table V), it was found that the aldehydes of higher molecular weight deposit some tar on the surfaces of the strip and test container. The amount of tar formed varied with the compound.

Besides the aldehydes, other known reducing agents were tried. All of the compounds listed in Table VI were somewhat effective, but not as efficient as glyoxal.

#### Conclusions

A definite need exists for a ferric ion reducing agent to lessen the corrosion of heat-exchange equipment during acid cleaning. Glyoxal, a dialdehyde, has been shown by laboratory tests to be an effective and economically feasible ferric ion reducing agent. Its use in acid cleaning a boiler has been successful, resulting in a saving of metal. Phenyl hydrazine is also a very efficient ferric ion reducing agent, but it is highly toxic and must be handled accordingly.

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# Electrochemical Factors In Underground Corrosion of Lead Cable Sheath\*

By V. J. Albano\*

**S**TRAY CURRENT is the principle cause of corrosion failures on underground telephone cables in most cities where trolleys are operated. To mitigate this condition, the cable sheaths are "drained" to the negative return system of the traction system. By this means not only is the stray current anodic area largely eliminated, but the cables automatically become negative to earth, and therefore are cathodically protected. The cathodic protection afforded in this manner prevents other types of corrosion from occurring. With the gradual abandonment of trolley systems, and with the extension of underground cables into non-trolley areas, the percentage of underground telephone plant receiving this protection is decreasing. As a result, the problems of lead corrosion due to such causes as galvanic and local cell action of various types, and chemical action by substances in the soil are becoming more prevalent. It is the purpose of this article to review some of the basic principles of corrosion not involving stray currents, and show how they apply to the problems of lead cable sheath corrosion.

The corrosion of lead in soils, like corrosion in any electrolytic medium, is the result of electrochemical action occurring on the surface of the metal. Differences in potential existing on the metal surface as a result of some chemical or physical inhomogeneity in the metal or its environment set up minute galvanic cells, and the current resulting from the operation of these cells causes corrosion.

The first quantitative expression for the relation between corrosion and current flow had its origin in the work of Michael Faraday in 1834. If, as is usually the case, corrosion is the only reaction which is occurring at the anode of a corrosion cell then, by Faraday's law, the rate of corrosion of the particular metal involved is directly proportional to the current flowing. That is

$$\frac{W}{T} = ZI \quad [1]$$

where  $W$  = weight of metal corroded in grams,

$T$  = time in seconds,

$I$  = current in amperes, and

$Z$  = a constant.

The constant  $Z$ , known as the electrochemical equivalent of the metal, is different for each metal. It is the amount of material dissolved or deposited during electroly-

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sis by the passage of one coulomb of electricity. Since it requires one Faraday (96,500 coulombs) to dissolve one chemical equivalent of a metal, then:

$$Z = \frac{\text{Atomic weight of the metal (in grams)}}{\text{Valence change} \times 96,500} \quad [2]$$

The value of  $Z$  for lead, when it corrodes to form bivalent lead ions, is  $1.074 \times 10^{-3}$  grams per coulomb.

By using equation [1] and knowing that lead has a density of 11.34 it can be calculated that if the lead were being corroded by a current of 0.1 milliampere operating over a surface one square inch in area, 0.018 inch of metal would be lost in one year.

In the above equation,  $I$  may be replaced by  $E/R$ , in accordance with Ohm's law, where  $E$  is the operating voltage of the corrosion cell and  $R$  is its overall circuit resistance; therefore

$$\frac{W}{T} = Z \frac{E}{R}$$

This equation applies to the case where only a simple two-electrode cell is operating. Actually, innumerable cells exist on the surface of a corroding metal, and the attendant mutual interaction among the electrodes renders a mathematical treatment of the problem quite difficult. However, the factors which affect  $E$  and  $R$  in the simplified case would affect, in the same manner, the corresponding quantities in any more generalized equation. While a more generalized treatment would be of theoretical interest, the feasibility of applying such a relationship quantitatively to cases involving soil corrosion is negligible because of the complexity and indeterminate na-

ture of the variables involved. The simplified equation is nevertheless a very useful qualitative guide to an understanding of the rate controlling factors.

Expressed in words instead of symbols, it may be said that the rate of corrosion of any given metal is directly proportional to the driving forces, or electromotive forces of the corrosion cells, and inversely proportional to their overall circuit resistance.

### Basis in EMF Series

The driving forces, or initial electromotive forces of the corrosion cells arise from chemical or physical inhomogeneities in the metal or its environment. These driving forces have their basis in the well-known emf. series which applies specifically to solutions in which the metal ion concentration is normal. The following are some of the more common causes of potential differences due to inhomogeneities in the metal:

- 1) Inclusions of more electro-positive (more noble) metals, or metal oxides.
- 2) Differential strains.
- 3) Differential grain sizes.
- 4) Differential orientation of crystal faces at the surface of the metal.

Of the four metal factors indicated, the first is the most important insofar as corrosion of lead cable is concerned. Inclusions of lead oxide may occur during the process of extruding lead sheath. If these inclusions happen to settle at the surface of the sheath, they will set up corrosion cells in which the adjacent lead is anodic to the oxide. Also lead which has tarnished or

become coated with a thin film of corrosion products will be cathodic in nature to clean, bright lead, and promote its corrosion. The potential set up under these conditions is sufficiently large to cause a corrosion hazard to exist for a new cable when it is bonded to an older cable, and run parallel to it in the underground conduit system. Inclusions in the lead may also occur as a result of metal particles becoming inadvertently embedded in the sheath during handling and installing operations. The corrosion cells thus initiated will be hazardous to the lead if the embedded metal is more noble than lead. Another possible source of contamination develops when the cable is immersed in an electrolyte containing dissolved salts of a more noble metal. Therefore, if the water in a manhole or duct contains dissolved copper, copper will be deposited on the lead, thereby setting up a lead-copper corrosion cell. The potential differences established in cells of the inclusion type may be of the order of 0.5 volt, which is quite appreciable. Because of the localized nature of such cells, the corrosion which occurs is of the pitting type, and is therefore more serious than uniformly distributed corrosion.

#### **Pitting Most Serious Type of Corrosion**

Potential differences due to differential strains are not likely to exist in lead since it is a soft metal and relieves itself of strains very readily. Differences in grain sizes, and orientation of surface crystals are responsible for the existence of numerous very small potential differences on the surface of the lead.

However, corrosion from cells of this type would be uniformly distributed, and therefore not so serious as corrosion of the pitting type.

#### **Environmental Factors**

Among the more important inhomogeneities in the environment which cause potential differences to appear on the surface of lead cable underground are:

- 1) Differences in electrolyte concentration.
- 2) Differences in electrolyte composition.
- 3) Differences in oxygen concentration.

Of these environmental factors, those involving corrosion cells created by differences in oxygen concentration are more prevalent than those due to metal ion differences, and they are capable of producing a much greater potential than cells of the metal ion difference type. In this latter type of cell, a potential difference of only 0.03 volt results in the case of lead in contact with two solutions whose lead ion concentrations differ tenfold, the specimen in the more dilute solution being anodic. In a differential oxygen cell using lead electrodes, potential differences of 0.1 volt have been measured. In this type of cell the metal area in contact with the electrolyte having the lower oxygen concentration is the anode.

When lead is immersed in an electrolyte which does not originally contain any lead ions, some lead ions will be formed the instant the lead comes in contact with the solution. The potential of the metal would then be determined by the concentration of the metal ions which form around it. Because the number of



ions produced depends on the nature and concentration of the electrolyte, it is not always possible in such cases to predict the effect on the electrode potential of variations in the concentration of the electrolyte. In many cases, the electrode in the more concentrated electrolyte will be anodic to the electrode in the dilute concentration. This is in direct contrast to the case where the electrolyte consists of a solution of ions of the metal involved. Shephard,<sup>1</sup> of the United States Department of Commerce, National Bureau of Standards, has found that a section of pipeline which passes through a soil of high conductivity (and therefore high electrolyte concentration) is anodic to a section which passes through an adjacent region where the soil is of low conductivity.

#### Aeration Cells Common Cause of Corrosion

Differential oxygen or aeration cells exist in many forms, and are a common and serious cause of corrosion of lead cable sheath. A large gob of inert matter saturated with electrolyte and resting on a cable constitutes a differential aeration cell in which the area beneath the gob is less accessible to the air, and consequently is anodic to the surrounding area. Coarse particles of sand, and drops of electrolyte in contact with the sheath may produce a similar result.

Differential aeration cells are also formed when small crevices exist on a cable. The region at the bottom of a crevice is anodic to the outer, more aerated area and consequently tends to corrode. For this same reason a corrosion pit, once started, tends to

be self-perpetuating, particularly if it contains corrosion products. Also, when a cable is partly immersed in a solution, corrosion may occur just below the water line because of the differential aeration cell existing there. The electrolyte at the interface between a solution and air is richer in oxygen than the solution directly below it, so that the metal area just below the water line is anodic to the metal just at the water line, and consequently will corrode. Differential aeration cells may also form when loose duct packing such as oakum is used around cables at duct openings.

#### Polarization and Working Voltage of Corrosion Cells

The inhomogeneities which have been discussed are the causes of the corrosion-cell voltage. However, during the operation of such a cell, the actual working voltage is less than the open-circuit voltage because of polarization which occurs at either or both the anode and cathode areas. Polarization is caused by electrochemical changes resulting in the accumulation of the products of electrolysis at the electrode-electrolyte interface as a consequence of current flow. It is in effect a counter-electromotive force which increases as the current increases. There are four sets of polarization conditions which may affect the working voltage of corrosion cells.<sup>2</sup> These are represented in Figure 1. In Cell 1, neither the anode nor the cathode is appreciably polarized and consequently the working voltage is high even at high current densities. Cells 2 and 3 show the conditions in which the decrease in working voltage is due al-

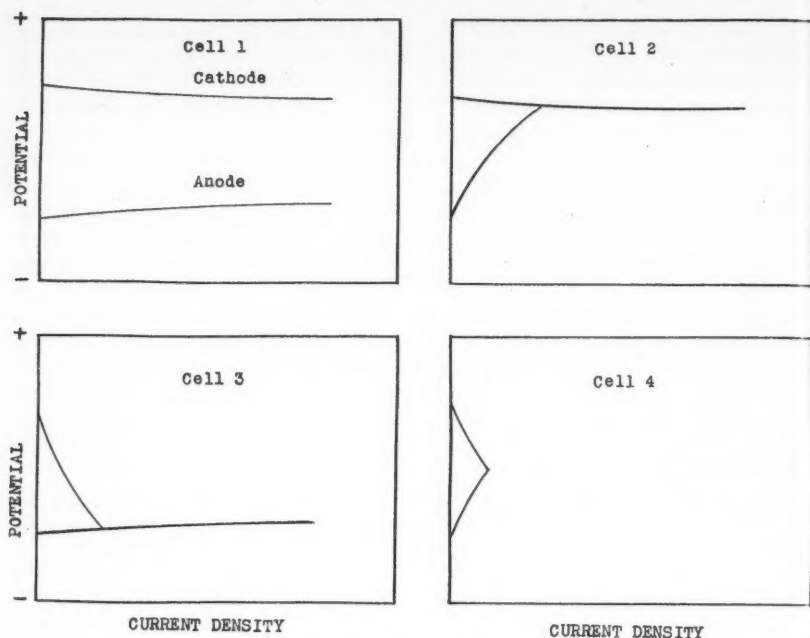


Figure 1—Types of polarization in corrosion cells.

most entirely to anode and cathode polarization, respectively. In Cell 4, the working voltage decreases very rapidly with increasing current density since both electrodes undergo marked polarization.

At the cathode areas, polarization may occur as a result of the accumulation of atomic hydrogen. The extra potential required to liberate the gas in molecular form is called the hydrogen overvoltage, and varies considerably with different metals. Since the hydrogen overvoltage on lead is about 0.5 volt, cathode polarization from this cause can be an important factor in reducing the current flow in corrosion cells. In the presence of oxygen or oxidizing agents, hydrogen ions are discharged

much more readily, thereby decreasing polarization and stimulating corrosion.

At the anode, the principal cause of polarization is the increase in metal ion concentration which takes place because of the slowness with which these ions migrate away. This higher metal ion concentration causes the electrode potential of the anode to become more electropositive (more noble) thus decreasing the working voltage of the corrosion cell and thereby retarding corrosion. If the electrolyte in which the anode is operating contains anions capable of forming insoluble compounds with the metal ions, precipitation of such compounds will occur as soon as their limits of solubility are

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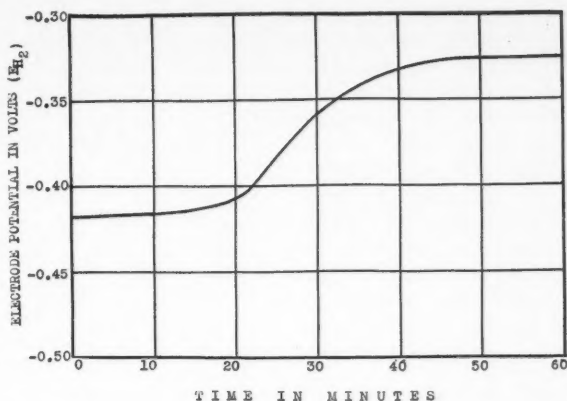
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reached. This precipitation may take place in such intimate contact with the anode areas that they become shielded from the corrosive environment and are consequently protected from further attack.

Lead withstands the corrosive action of soils mainly because of the presence therein of numerous film-forming substances. Thus silicates, sulfates and carbonates form insoluble films over the anode areas and tend to shield them from further attack. After these anode areas have been protected, other areas which were less anodic, in turn become covered with a film until eventually all but the small residual cathodic areas are covered.

The results of a laboratory experiment showing the progress of the formation of a sulfate film on a specimen of lead-antimony (1.0 percent) cable sheath alloy when partially immersed in a normal sodium sulfate solution are shown in Figure 2. The break in the initial potential of lead after about 20 minutes indicates that sulfation is approaching completion. At the end of 40 minutes the process is practically complete, and the potential measured is that of the residual cathode areas.

The effect of silicates in preventing the corrosion of lead is especially notable. As little as 10 parts per million of sodium silicate will reduce tenfold the corrosiveness of distilled



Lead-antimony cable sheath alloy in normal sodium sulfate.  
Figure 2—Sulfation-time curve.

water. The illustrations in Figure 3 show that New York City tap water, which contains only about 40 parts per million of dissolved substance, is hardly corrosive to lead, whereas distilled water, which lacks film-forming substances, corrodes lead very readily.

#### Silicates Most Effective

The protective effect of sulfates is not so pronounced as that of silicates, probably because the greater solubility of lead sulfate results in a more porous film. Soil carbonate also exercises a protective action similar to silicates and sulfates, but its effectiveness is reduced because of the solvent action of carbon dioxide, which usually exists in the underground cable plant in higher concentrations than in the atmosphere.

Film formation may also occur on the cathode areas of underground cables as a result of the precipitation on these areas of colloidal particles of silt, clay or organic matter which exist in the soil. This deposition occurs by cataphoretic action in which

the positively charged colloidal particles migrate to the cathode areas, become discharged and deposit on the sheath. Calcium bicarbonate, if present in the soil electrolyte, may cause calcium carbonate film formation over the cathode areas. The cathodic reaction first produces calcium hydroxide, which then interacts with the bicarbonate and precipitates a film of calcium carbonate on the cathode areas.

### Not All Soil Protective

All soil constituents do not have a protective action on lead. There are some which cause serious acceleration of corrosion. Of these oxygen, nitrates, organic acids, and alkalis are the most often encountered.

Oxygen and nitrates act as cathode depolarizers by facilitating the discharge of hydrogen ions at the cathode areas. By this action, an increase in the working voltage of the corrosion cells is caused, resulting in an increased rate of corrosion. While the depolarizing action accounts for the increase in corrosion, it is very often the physical process of getting the depolarizing agents to the electrodes that determines the rate at which depolarization will occur. In this connection, the texture of the soil is an important factor, because soils of coarse texture, such as sands, will allow more rapid penetration of air and soil electrolyte to the metal, and thus stimulate corrosion.

Organic acids are capable of causing serious corrosion of lead. Acetic acid is sometimes encountered in underground cable plant, since it may be exuded from certain types of wood conduit if they are subjected

to excessive heat, particularly in the presence of moisture.<sup>3</sup> Although a weak acid, it is capable of causing serious damage to lead cable, particularly in the presence of oxygen and carbon dioxide. The oxygen which is present in the environment promotes corrosion by depolarizing the cathode areas. At the same time carbon dioxide, which invariably exists in the underground plant, reacts with the lead acetate formed at the anode areas to produce lead carbonate, which is insoluble. The lead carbonate so produced does not form

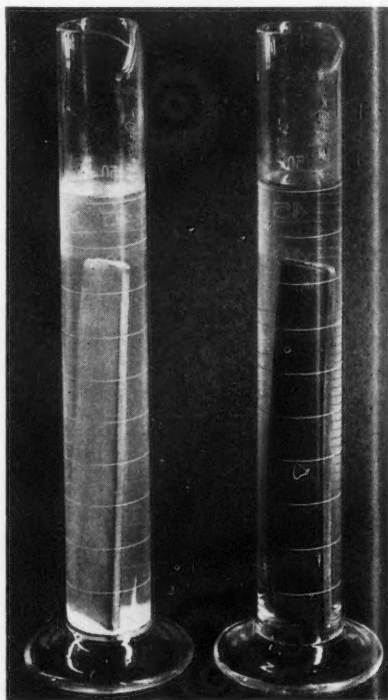


Figure 3. Effects of silicates in preventing corrosion of lead. Left, lead in distilled water. Right, New York tap water containing about 44 ppm of dissolved substances.

as a film in close contact with the lead and consequently is not protective. The carbon dioxide, in this role, acts as an anode depolarizer, because it tends to maintain a low concentration of lead ions around the anode. Because of the depolarizing action of these two gases, corrosion will proceed at a rapid rate. In addition to depolarizing the anode, the carbon dioxide, in reacting with the lead acetate, regenerates acetic acid, which can again attack the lead. Because this cycle can continue indefinitely, a small amount of acetic acid can cause extensive cable corrosion.

### Formation of Alkali

Lead is a metal which is subject to attack by strong alkalis as well as acids. In the underground cable plant, alkali may be formed during periods when the cables are excessively negative to earth, or may be encountered when "green" concrete is in the vicinity of the cables. In either case, corrosion cells are set up in which sodium or calcium hydroxide constitutes the electrolyte. The course of the corrosive action will depend to a great extent upon the arrangement of the components of the corrosion cell. Thus if the cable is completely surrounded by electrolyte, uniform attack of the sheath will occur, since in this case corrosion is the result of the operation of innumerable minute cells on the surface of the metal. Under these conditions, lead goes into solution as sodium or calcium plumbite. When the solution adjacent to the lead becomes saturated with these compounds, lead monoxide will begin to crystallize out in colors varying from yellow to orange to red.

Lead carbonate will form also because of the concurrent action of carbon dioxide on the plumbite solutions and on the lead monoxide. In the field, the conditions encountered most often are those in which portions of the cable are in contact with moist duct or rest in shallow pools of stagnant electrolyte. By this arrangement, concentration cells, or differential aeration cells are established in which the anodic areas will undergo severe corrosion in the presence of the alkaline electrolyte. Even the cathodic areas of these cells may be subject to slow attack by the alkali which accumulates there. Corrosion in these areas is usually widespread but not deep, and will possess the characteristic orange-red colored lead monoxide corrosion products. Figure 4 shows the differential aeration type of attack which occurred when glass beads moistened with sodium hydroxide rested on a lead plate. The areas just outside the area of contact of the beads and lead are cathodic and are covered with the red form of lead monoxide.

The actual corrosion of lead in soils is rarely a single, clearly defined process. Several reactions may occur either simultaneously or consecutively, and the net effect will depend on the nature and relative amounts of the various reacting constituents.

For convenience, the soil constituents involved in corrosion may be classified either as protective or corrosive agents. The protective agents are usually those substances which polarize the electrodes or which form films on them. The corrosion promoting agents are usually those substances which depolarize the electrodes or which form soluble corro-

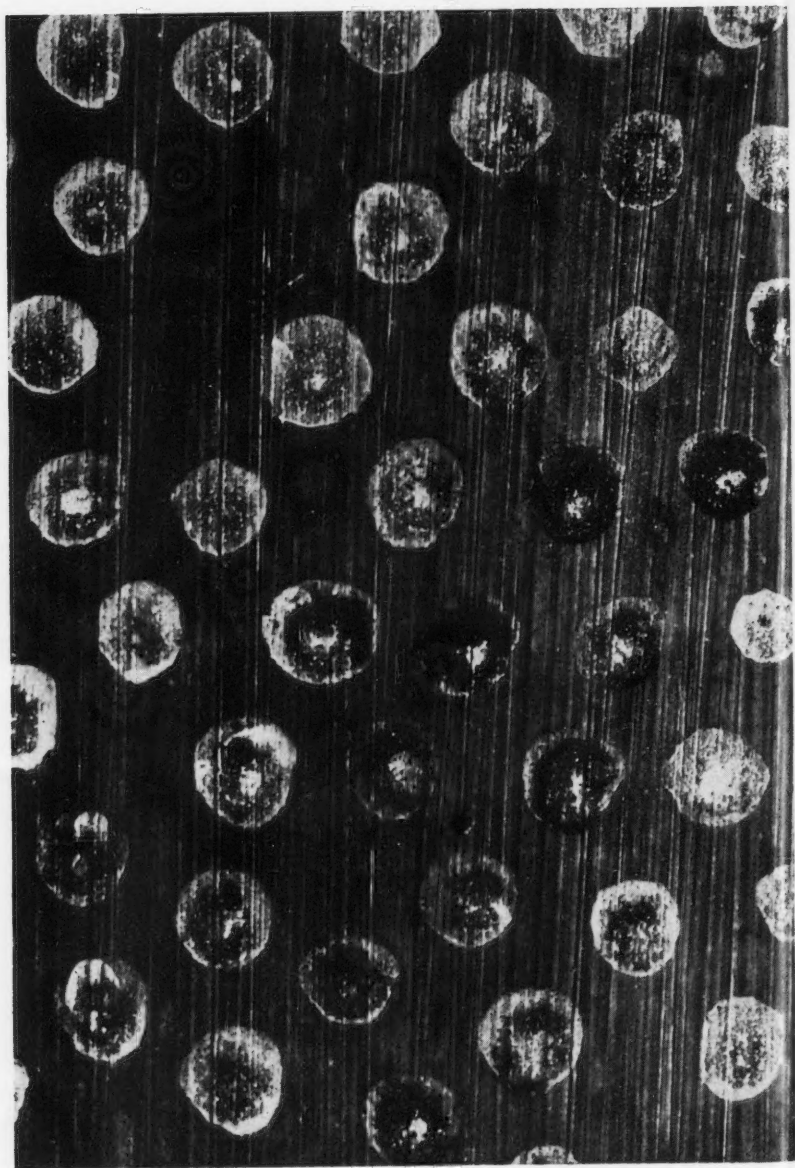


Figure 4—Corrosion of lead in contact with glass beads moistened with sodium hydroxide.

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sion products with the metal. It is the ratio of the concentration of protecting agents to corroding agents which determines the rate at which corrosion will occur and also the character of the attack. If this ratio is high, the lead sheath will be protected; if it is low, corrosion will occur and will probably be of a uniform nature. Intermediate values of this ratio indicate that only partial protection is afforded by the film-forming agents, and consequently corrosion is likely to be of a localized nature resulting in a pitting type of attack.

### Factors Involved

The balance between polarization and depolarization of the anodes and cathodes of corrosion cells determines their working voltage, but this factor alone does not determine the rate of corrosion. The resistance of the internal and external circuits of the cells involved must also be taken into consideration since these constitute the obstructions to the flow of current. The external resistance is the short metallic path between the anode and cathode areas and is usually so low compared to the other parts of the circuit that it does not have any controlling influence on the current flow. The internal resistance is composed of the resistance of the electrolytic path between anode and cathode areas plus the resistance of any protective film existing at either electrode. If no film exists, then the current flow is determined solely by the resistance of the electrolyte. When protective films do exist, their resistance is usually so high compared to the other parts of the circuit that they exercise almost

complete control over the corrosion current.

### Ideal Situation

The complete prevention of corrosion would require the eradication of all corrosion cells from the surface of the metal. This could be accomplished if it were possible to remove all heterogeneities from both the metal and its environment. Spectroscopically pure zinc is resistant to attack by pure hydrochloric acid for this reason. While such an ideal set of conditions cannot be expected in actual practice, still most of the protective measures which have been devised owe their effectiveness to the application of the principle of preventing the formation and operation of corrosion cells.

### Keep Out Moisture

Because an electrolyte is absolutely essential for the operation of a cell, it is possible to eliminate corrosion of underground lead cables by keeping the environment dry. It is usually not feasible to maintain the surroundings completely dry, but the environment immediately adjacent to the lead sheath could be kept free from electrolyte by the use of impervious coatings of rubber or plastic materials. This method of protection is used where the surroundings are extremely corrosive. The use of coatings which are not impervious to water also help reduce corrosion by maintaining a uniform electrolytic medium around the cable sheath, thus tending to eliminate corrosion cells caused by heterogeneity of the environment.

A reduction in corrosion can also be effected by altering the environment so that the ratio of protecting

agents to corroding agents is increased. This may be done by adding film-forming substances to the environment in amounts sufficient to maintain an adequate supply at all times. Needless to say, if the elimination of corroding agents is possible, this should be the first step taken to obtain a more favorable ratio of protecting agents to corroding agents.

The cathodic protection method of coping with corrosion is at present receiving widespread attention. It depends upon making the cable

cathodic with respect to its surroundings. Protection is obtained by this means when the cathodes of the corrosion cells are polarized to the open-circuit potential of the anodes.<sup>4</sup> Under these conditions no corrosion current can flow, since no potential difference exists at the metal surface. In using this method for protecting lead cables, the sheath must not be made excessively negative to earth since the alkali which forms under these conditions may be capable of attacking the cable.

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### Discussion

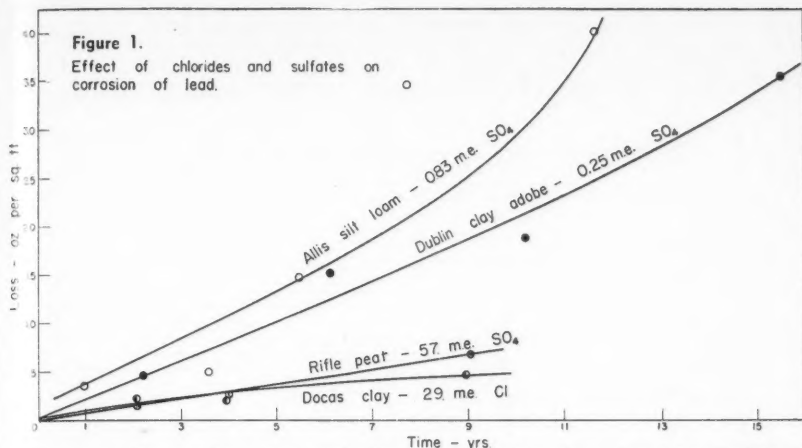
By I. A. Denison\*

The constituents of soils which accelerate the corrosion rate of lead are considered by the author to be oxygen, nitrates and organic acids. Under special conditions, as in the case of overprotection cathodically, the alkali produced on the lead surface may, of course, cause serious corrosion. On the other hand, the soluble materials in soils which tend to inhibit the corrosion of lead by

causing the formation of difficultly-soluble films are considered to be silicates, sulfates and carbonates. Films of calcium carbonate formed by precipitation of calcium and bicarbonate ions on the local cathodes likewise tend to inhibit corrosion, probably by partially excluding oxygen from the cathodic areas.

The data which have been obtained by the National Bureau of Standards in the course of its extensive investigation of corrosion in

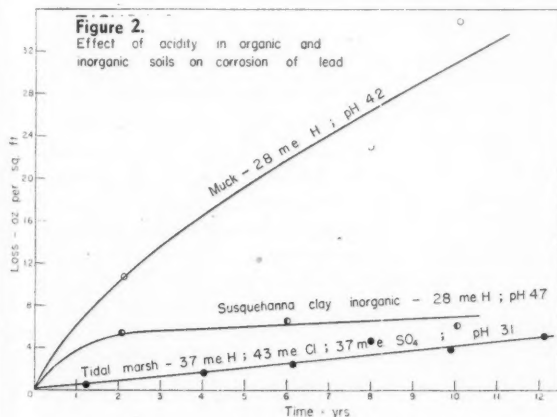
\* Underground Corrosion Section, National Bureau of Standards, Washington, D. C.

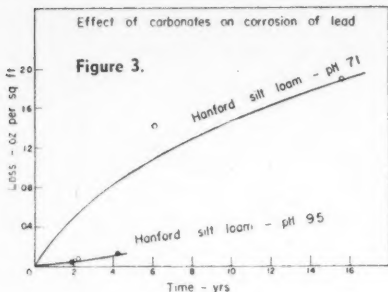


soils generally confirm the author's conclusions concerning the effect of various environmental conditions on the corrosion of lead underground. For example, acceleration of the corrosion of lead by organic acids in soils and inhibition of corrosion by sulfate ions have been observed at a number of the Bureau test sites. These effects are illustrated in Figure 1, in which the corrosion losses on specimens of lead cable sheath removed from several test sites are

shown plotted against the period of exposure. The high corrosion rate of approximately 0.3 oz/sq ft/yr for the muck soil is certainly to be ascribed to its high total acidity of 28- milligram-equivalent of hydrogen ion per 100 grams of soil; while the very low rate of 0.04 oz/sq ft/yr in the tidal marsh, containing even a greater amount of ionized hydrogen and having a lower pH value, no doubt resulted from the marked inhibiting effect of high concentrations of sul-

fate and chloride ions. That inorganic acids derived from the weathering of aluminosilicates are relatively ineffective in accelerating the corrosion of lead is shown by the curve for the Susquehanna clay. The corrosion data shown graphically in Figure 2 likewise illustrate the marked inhibiting effect conferred by

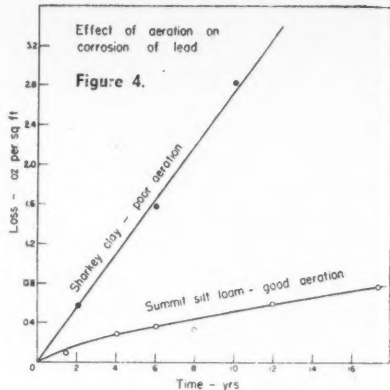




high concentrations of chloride and sulfate ions in soils.

The inhibiting effect of carbonate ions on the corrosion of lead in soils is illustrated by loss of weight-time curves for specimens of lead cable sheath removed from two test sites in the Hanford silt loam (Figure 3). The presence and absence of alkali carbonate at the two sites is indicated, of course, by the pH values of 9.5 and 7.1. Although data for four years only were obtained from the alkali-containing soil, there is no doubt that the corrosion rate in this site was greatly reduced by the alkali present.

Albano properly emphasizes the importance of differential aeration as a factor in the corrosion of lead underground. However, his inference relative to the effect of soil texture on the rate of corrosion of lead is not borne out by the Bureau data. In fact, these data, considered as a whole, lead to the opposite conclusion—namely, that as with ferrous metals, the corrosion of lead is greater in poorly aerated soils than in well aerated soils. In Figure 4, for example, the corrosion of lead is seen to be severe in the very poorly



aerated Sharkey clay, but relatively slight in the well aerated Summit silt loam. The curves for the muck soil (Figure 1) and for Allis silt loam and the Dublin clay adobe, (Figure 2), also illustrate the corrosiveness of poorly aerated soils with respect to lead. As long as the rate of corrosion of lead is determined by the rate at which the cathodic reaction proceeds, it necessarily follows that the more permeable the soil to air, the higher will be the corrosion rate. But oxygen in excess of that required for depolarization of the cathode apparently exerts an inhibiting effect on the anode reaction, with the result that the overall corrosion rate is low. Here again the analogy between the corrosion of lead and the corrosion of ferrous metals with respect to differential aeration may be noted. The fact that severe corrosion of ferrous metals is most generally associated with poor aeration indicates that oxygen, while accelerating the corrosion rate at the local cathodes, has an inhibiting effect at the anodes.

# Principles of Immersion and Humidity Testing of Metal Protective Paints\*

By A. C. Elm\*

PERHAPS the most difficult task confronting the paint test engineer is the determination of the "durability" of an organic protective coating, that is, the protection it affords under conditions of actual service. The majority of painted articles are subjected to a wide variety of external influences which may vary over a relatively wide range so that it becomes very difficult to study the effect produced by each individual factor, or by any combination or sequence of factors, and to correlate the results into a reliable picture of the value of the paint as a protective medium.

One solution of this problem consists of actual service tests on an extended scale and the application of statistical methods to the exposure results. In this case, no particular effort need be made at controlling or studying the effect produced by all variables individually, but a sufficiently large number of tests must be conducted so that the final average values arrived at will have taken into consideration all kinds, degrees, and combinations of variable conditions. Despite its many advantages,

actual service testing, however, cannot always be employed for several reasons, the most important of which is lack of time and suitable test surfaces. Therefore, the paint test engineer is forced to resort to less time-consuming laboratory methods and for this reason he is greatly interested in establishing the reproducibility and validity of laboratory tests.

There is no proved or even generally accepted laboratory test procedure which would make it possible to state unequivocally that a given paint will or will not serve the purpose for which it is intended, and it is frequently necessary to design special testing equipment and procedures. This, however, appears to be a much more difficult and time-consuming undertaking than it should be, largely because the basic principles of laboratory testing are not fully appreciated. An analysis of some of these principles, therefore, may prove to be helpful and timely.

A discussion of the application of these principles to paint testing in general would lead too far and would consume much more space than has been allotted to this article. Therefore, this discussion will be aimed particularly at an analysis of the testing of metal protective paints by ex-

\* Reprinted by permission from ASTM Bulletin, 142, 9-27, Oct. (1948).

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posure to high humidity or immersion in suitable aqueous solutions, although much of this report may be applicable to paint testing in general.

### Designing a Test

In designing a test procedure or equipment there are three distinct steps: (1) the formulation of a theory of what is needed, (2) the actual development of an instrument or technique, or both, and (3) the extensive use of this instrument or procedure to demonstrate its parallelism with actual service experience. The first of these steps is not only the first chronologically but also the first in importance. It is a waste of time and effort to design testing equipment or procedures before the problem has been fully defined and its aims clearly fixed. The necessity of diagnosing the problem correctly and working it out in considerable detail before entering upon steps (2) and (3) cannot be overemphasized.

The purpose of humidity and immersion testing of metal protective paints is to compare various paint systems for their ability to protect metals against corrosion and destruction under service conditions where water or aqueous solutions are the principal deteriorating agents. For tests of this sort, paint films must be tested, firmly attached to a suitable substratum, and their relative abilities to perform their functions measured by the changes taking place in the substratum as a direct result of the exposure to the test conditions.

To avoid misleading results, it is essential that any significant changes in the appearance of the test specimen observed in the course of the

test be the direct result and a quantitative measure of the failure of the coating to protect the substrate adequately, and not the accidental result of any differences in the substrate, or unknown or uncontrolled variations in the environmental conditions.

For example, when paint systems are to be compared for their ability to protect steel against rusting, care must be taken that any differences in rusting observed are a measure of differences in the protective coatings under test and are not due to differences in the corrosivity of the substrates or to some other external factor.

### Control of Substrate Uniformity

The substrate materials commonly available for paint tests of this sort are not uniform. Not only do members of a given panel series differ, but significant areas on the same panel may differ sufficiently to cause marked differences in the behavior of paint films. Therefore, to insure the reliability of panel tests for the quality of metal protective paints, the substrate specimens must be so selected and prepared as to guarantee (1) that each panel of a given test series is uniform in itself, and (2) that all members of the series, immediately prior to the application of the test paints, are as nearly as possible identical in behavior and properties.

In order to determine how this might be accomplished, let us look more closely at what is to be accomplished. It is desired to find a procedure which will permit the determination of the relative ability of a given series of paints to prevent the corrosion of steel objects exposed to



high humidity or immersed in water or aqueous solutions.

The surfaces on which these paints are to be tested, therefore, should, in the absence of any protection, that is, when exposed in the unpainted condition, corrode at a uniform rate and to the same degree. Each individual member of the series should corrode uniformly all over its surface and should be as free as possible from pitting and other localized corrosion effects, and all members of the series should be alike in these respects. Any preparative treatment given the test panels should be for the sole purpose of making sure that this condition prevails.

It has been proposed that, in order to determine whether steel panels have been properly prepared, they be tested for their ability to support a film of water without break. Actually, such a test serves only to indicate whether the panel surface is wetted uniformly by water. It does in no way guarantee that the same surface will also be just as uniformly wetted by the paint to be applied to it, much less would it indicate that the panels are identical in their response to failures of the test paints. What is needed is not necessarily a fat-free, rust-free, smooth or polished test surface but one that will promptly, reliably, and reproducibly indicate by visible rusting whenever a paint film fails to protect it against corrosive influences.

A test which may be useful in this connection was proposed by Mears.<sup>1</sup> A panel specimen to be tested for uniformity and corrosivity is dotted with a large number of droplets of a suitable corrosive liquid, and the time of the first appearance of cor-

rosion in each droplet as well as the nature of the corrosion product are noted. The average time elapsed to the first appearance of rust serves as a measure of the corrosivity, while the nature of the corrosion product and the spread between these values for the various droplets measures uniformity. Hydrogen peroxide is a suitable testing liquid. It is unfortunate that this test spoils the panel for use in any paint test without further treatment. Although we have not yet been able to do enough work with this test to establish definitely its reliability, we are confident that it will be of considerable help in the development of a procedure for pre-treating paint test panels and as a routine control test for panel preparation.

For obvious reasons it is desirable to obtain results of practical significance in the shortest possible time. Therefore, it is not desirable to have the steel surfaces under the test films in a passive or only slightly active condition; on the contrary, it is preferable to have them in a condition of high activity or corrosivity, so that they respond readily to any failure of the test paint system to exclude corrosive agents. On the other hand, it does not seem desirable to aim at the highest possible degree of activity since in all practical cases some time must elapse between surface preparation and the application of the first coat of paint, and corrosion of the test surface during this interval should be avoided. Therefore, it would seem preferable to aim at some intermediate degree of corrosivity which possesses sufficient stability to allow application of test paint.

### Effect of Panel Size

When paints are subjected to laboratory performance tests, it is usually necessary to reduce the size of the test area to a very small fraction of the area encountered under actual service conditions. This means that corners and edges occupy a relatively large portion of the test area. Therefore, corner and edge effects will be out of proportion to those usually encountered in actual service unless special precautions are taken to prevent an excessive influence of corners and edges upon the test results. Such effects are absent or greatly minimized on spherical or cylindrical test specimens of sufficient diameter.

The mechanical difficulties encountered in preparing and handling such specimens, however, make them unsuitable for routine testing of metal protective paints. A practice of minimizing edge and corner effects which has been widely used consists of protecting all corners and edges with additional coats of paint or several coats of wax. This practice has much to commend it and may be expected to give satisfactory results provided the original panel area is not so small that the paint or wax edgings reduce it to a size inadequate for the purpose in mind.

There seems to be little doubt that for the sake of uniformity of any one panel of a set it is necessary to avoid coarse contours and large grain size. This takes on added importance as the panel size is reduced. It seems futile to specify in detail the degree of smoothness needed, for it depends much on the consistency characteristics of the test paint and its ability to fill cracks and crevices, but it

is readily appreciated that it should be of such a degree as to guarantee a continuous coating of adequate thickness when the test paint is applied according to the procedure specified.

### Effect of Film Imperfections

The objection may be voiced that test panels prepared with such care bear no relation to actual service experience and therefore are of little, if any, practical value. It has been often stated that there is hardly any paint job absolutely free from defects and scratches and that therefore the results obtained with imperfect panels were of much greater practical significance than any results obtained with perfect panels. In fact, many paint test engineers favor deliberately scratching through the paint film to determine the resistance of the test paint to loss of adhesion and rust creepage from a bare area. There is no objection to such practices provided the results obtained with them are viewed from the proper perspective.

Such a test does furnish valuable indications regarding the protection afforded by a paint system under a highly specific set of conditions. It must be remembered, however, that the results of such a test are significant only if the substrate areas bared in this way on the various specimens of a test series possess identical activity. This presupposes that the panels were originally of the same activity and that the scratch actually penetrates to and lays bare an area of active metal. In addition care should be taken that the ratio of damaged to undamaged area is not too far out of proportion to that usually encountered in actual prac-

tice, or, if this cannot be avoided, that this factor is at least taken into consideration when the results are interpreted in terms of service expectancy. When an actual paint job, such as on a ship, is examined and rated, the effect of film damage automatically appears in its proper perspective, whereas on a panel deliberately scratched it is frequently so greatly out of proportion that it may completely upset the balance of the test results.

As far as the application of the test paint is concerned, it does not really make much difference what method is chosen as long as care is taken that the coating is continuous and of uniform thickness. Kittelberger<sup>2</sup> has shown that in immersion testing, pin holes and exposed freely corroding areas of metal in electrical contact with the test specimen may exert a marked influence upon the type and rate of failure suffered by the test paint.

It is obvious then that the effective film thickness is the thickness of the coating at the thinnest point. Therefore, methods of determining the film thickness of a coating by calculation from its weight and specific gravity may be very misleading. They make no allowance for either the uniformity of the coating or the roughness of the substrate surface. Continuity measurements, such as those proposed by Young and Gerhardt<sup>3</sup> are very useful in determining the minimum coating thickness needed to produce a continuous coating on a given surface and therefore serve as a measure of the roughness of the substrate.

### Selecting a Testing Technique

Perhaps the most controversial

part of any specification for determining the protection afforded a metallic substrate by a paint system is the testing technique. While some advocate total immersion in water or aqueous solutions, others swear by salt spray or moisture condensation tests. Without advocating either procedure, we should like to describe some observations which we made in the course of extensive experiments and which throw some light upon the principles involved in the absorption of water by paint coatings, their blistering and failure to prevent rusting.

Water passes through the face of a paint film under the influence of two forces, osmosis and electroendosmosis. For the purpose of this discussion, osmosis is defined as, the transfer of water through a membrane under the influence of a solute concentration gradient, while electroendosmosis is the transfer of water through a membrane under the influence of an electrical potential gradient.

In the absence of endosmotic influences, water-soluble material within a paint film, which may be of inorganic or organic nature, causes the formation of an osmosis cell, with the paint film itself playing the role of the semipermeable membrane. Water is pulled into the film and may create enough pressure within or under the film to cause blistering.

If this is correct, paint films should absorb less water and blister less in salt water than in distilled water, for the salt in the bath will compete for the water molecules, and the forces which cause the water to penetrate the face of the film no

longer will be the osmotic pressure resulting from the presence of the water-soluble material within the film, but will be this osmotic pressure minus the osmotic pressure of the salt solution constituting the bath.

That this is so is readily demonstrated. Apply any given paint—but preferably one of relatively poor water resistance—to several glass panels, and, after allowing them to dry thoroughly, immerse the panels in solutions ranging in salt content from zero to that of sea water (about 3.5 percent). A periodic check of the weight increase of these panels will reveal that it is an inverse function of the salt concentration of the bath. The effect of this water absorption becomes visible in a short time. While the paint films immersed in the distilled water will blister, the paint immersed in the 3.5 percent salt solution will remain free of blisters for many months. We have had four glass panels coated with four raw linseed oil primers pigmented with 30 percent by volume of zinc yellow, ZTO chromate, red lead, and iron oxide, respectively, immersed in a 3.5 percent salt solution for almost five years, and the last inspection several months ago showed them to be still free of blisters.

Some may question the validity of this statement, for no doubt they have often seen painted steel panels blister much more severely and rapidly in salt water than in fresh water. What has been said so far regarding blistering applies in this simple form only so long as there is no corrosion. But the suggestion was that glass panels be used to demonstrate the difference between

blistering in distilled water and in salt solutions. When a corrodible substrate is used, the blistering mechanism is a bit more complicated, for in the absence of corrosion, the blistering mechanism on a corrodible substratum is identical with that on a noncorrodible panel material. However, just as soon as corrosion sets in, the corrosion current flowing between cathodic and anodic areas will superimpose its influence upon that of osmosis, and blistering will be accelerated and intensified.

Extensive experiments conducted in our laboratories over the last three years have shown that over 90 percent of the water absorbed by a painted steel panel, coupled to a bare freely corroding steel panel of the same size and immersed in the same bath, passed through the face of the film as the result of electroendosmotic influences. The results of our tests highlight the tremendous influence exerted upon water absorption and blistering by electroendosmotic influences which may result from corrosion at pin holes and weak spots in the test film. They emphasize again the necessity of taking the utmost care in the preparation of the test specimens if interpretable results are to be obtained. In addition, they furnish the basis for a plausible explanation of some of the major difference in opinion existing among test engineers with respect to immersion and humidity test methods.

#### Humidity Versus Immersion Tests

On the basis of our results, some would expect that the amount of water absorbed by a paint film decreases as the vapor pressure of the immersion bath decreases, and that

the amount of water absorbed from a vapor would be the same as that absorbed from a solution of the same vapor pressure. In other words, the amount of water present in a paint film at the point of saturation or equilibrium with its environment would be the same regardless of whether the paint film was immersed in the solution or suspended in the vapor above this solution. It has been possible to demonstrate that this deduction holds, but it was found that the rate of water absorption by a paint film is appreciably greater when the film is immersed in the solution than when it is suspended in the vapor above the solution. That means that an immersed panel reaches the point of saturation or equilibrium faster than a duplicate panel suspended in the vapor. Of course, as stated above, this conclusion is valid only in the absence of endosmotic influences, which may materially modify the net result observed.

If this reasoning is carried to a logical conclusion, there should be no difference in the amount of water absorbed by a paint film immersed in distilled water or suspended above a water reservoir under conditions of a continuous condensation. Many may state that they have observed very marked differences. Although we have not yet had an opportunity to test this experimentally we venture to predict that the differences observed probably will be readily accounted for by temperature differences between the bath and the vapor. The chances are that the vapor itself and the panel suspended in it were close to 100° C., while the bath used in the comparison test was

most likely kept at room temperature. Doty, Aiken and Mark<sup>4</sup> were able to show that the moisture permeability of a high polymer film doubles with a temperature rise of about 10° C. It would seem reasonable to assume that a similar temperature effect is operative in the case under discussion here. Therefore, we believe that a paint system will absorb the same amount of water regardless of whether it is suspended in water vapor under conditions of continuous condensation, or immersed in distilled water at the same temperature. If this relationship could be proved experimentally some testing procedures could probably be simplified considerably. Humidity cabinets, which are meeting with so much favor among paint test engineers, are difficult to maintain and to control. It would seem simpler to replace such cabinets with immersion baths of comparable temperature and vapor pressure.

This applies with particular emphasis to the salt-spray test. The use of the salt solution decreases the water absorption of the paint film due to osmosis, and hence decreases markedly the rate of blistering as long as the film remains unbroken. Just as soon as the film is ruptured, however, electroendosmotic influences will predominate, and both the water absorption by the film and the corrosion of the bared substrate will proceed at an accelerated rate. But we are unable to see in what respect a salt-spray test offers any significant advantages over a simple immersion test in a bath of the same vapor pressure and the same temperature.

The second step referred to earlier,



the actual development of an instrument or technique or both, is nothing more than a logical application of the basic principles developed in the first step. It involves merely the detailed description of the techniques to be used in the preparation of the substrate material, the application of the test paint, and the testing of the specimen to insure that the conditions indicated to be necessary in the first step actually prevail. This phase of paint testing is too well known to require further discussion at this time.

### Translation to Practice

However, in many cases, the third of the three steps listed, the widespread use of the testing method to establish its parallelism with actual service experience, is not fully appreciated and will therefore be discussed at some length. As a disinterested observer, I have frequently been struck by the lack of agreement among experienced paint testing engineers regarding the practical value of test results and especially about their interpretation in terms of actual service, and I have gained the impression that many of the differences could be readily resolved.

Let us assume that reproducible and significant results have been obtained in a number of laboratory tests. The question, then, is what do these results signify in terms of length of life and protection the paint system offers under conditions of actual service? In the twenty years that I have spent in the paint industry, I have come to the conclusion that there is no substitute for actual experimentation and experience when it comes to translating laboratory test results into terms of

durability. The problem is the evaluation of the test method and the only way this can be done is to subject a sufficient number of paint systems simultaneously to the laboratory test and to actual service tests until a sufficiently large mass of related data has accumulated to serve as a basis for deriving a translation factor.

Many disagreements between paint testing engineers may be traced to the fact that each one has his own idea regarding the practical value or durability of the so-called standard paint systems. On closer analysis it will usually be found that the disagreeing paint testing engineers have a fixed mental picture of the value of the standard of comparison derived from a very few paint service tests, or experiences which happen to have made a lasting impression upon their memories. In my opinion, it is the major problem of the third or proving-in stage of the development of any standard test apparatus or procedure to fix the true value of the standard comparison paints.

The magnitude of this job becomes apparent when it is fully realized that in most service tests it is impossible to control either the nature and the preparation of the substrate or the conditions of application and service within the limits necessary for reproducibility within even tolerable limits. As a result of this condition, any one service test may rate a given paint good, intermediate, or bad depending on a more or less accidental combination of factors. Therefore, it will be necessary to seek a solution for this problem on a statistical basis. On the



face of it, this seems like a Herculean task doomed to failure before it is begun. However, similar and possibly more difficult, problems have been solved successfully.

The similarity between the problem of predicting the life expectancy of a human being on the basis of mortality studies and the problem of predicting the life expectancy of a paint film on the basis of service tests is apparent. A study of life insurance methods and an attempt to apply the laws of probability to paint testing may prove quite helpful.

Although unable to predict accurately the probable life expectancy of an individual, a life insurance company can predict with reasonable accuracy the average length of life under predetermined conditions of a certain class of people characterized by certain physical makeup,

certain habits, and certain environment. This exactly is the basic idea behind service testing: namely, to predict the average length of life of a certain type of paint under known conditions of exposure or use.

After suitable standard paints have been selected on the basis of extensive service tests, it is only necessary to include several specimens of each in every test series to be able to grade the experimental paints as to their probable life expectancy and performance value.

All of this may seem like a lot of trouble for a paint test, but really it is no more trouble than trying to interpret some of the results obtained when these precautions are ignored. Our experience has convinced us that efforts to make certain observations made are really a function of some inherent property of the test paint are well worthwhile.

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### Round Table Discussion

By W. W. Cranmer<sup>1</sup>  
(prepared discussion)

During May, 1945, our laboratory placed 200 panels on tide range exposure at Miami, Fla. These panels were in 20 groups, with 10 presum-

ably identical panels in each group.

Figure 1 illustrates the variation in rusting shown by one of these groups after three months' tide range exposure. Figures 2, 3, 4, and 5 show other groups from the same series. Figure 6 is identical with Figure 5, but shows the group after a six-months' exposure. Failure is

<sup>1</sup> Industrial Test Laboratory, Philadelphia Naval Shipyard, Philadelphia, Pa.

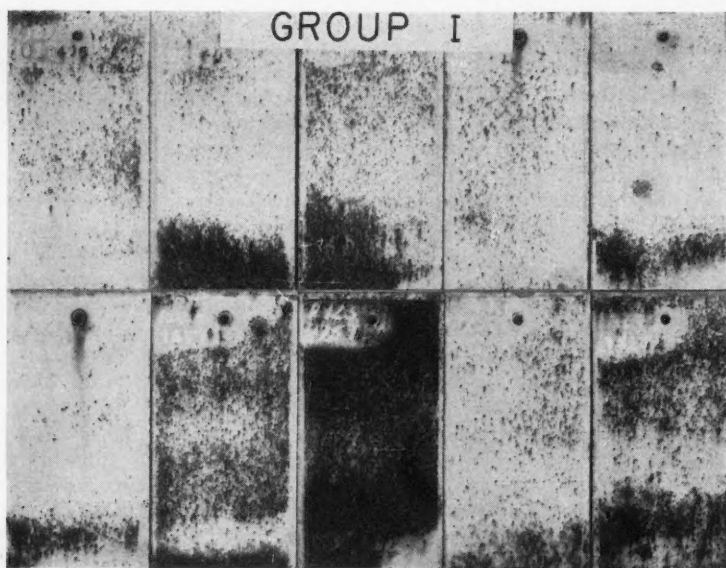


Figure 1—Three months' tide range exposure of ten duplicate panels.

more extensive, but the variation in results is of the same order.

Figure 7 shows four of the groups arranged for comparison. By selection of individual panels, it would be possible to rate any one of the four groups, in turn, as best.

In the case under discussion, the decision to expose 10 duplicate panels was made after consultation with a statistician, who indicated that the number would depend on the expected variability of results and the

discrimination desired. As to variability, we were prepared to state only that embarrassing discrepancies had sometimes been noted in tide range exposure and that exposure of a single coat, as necessary in this case, was known to be erratic. As to discrimination, we were again able to state only that we wished to distinguish clearly among systems, despite the anticipation that some would be nearly equivalent.

With such information, the correct answer is to expose an infinite number. This being impossible, the decision must be to expose as many panels as is feasible under the circumstances. Thus the original decision to expose 10 like panels may be described as a "guesstimate."

Having performed the work, however, the data now permit more

**TABLE I**  
**Chance Differences Between Average**  
**Rust Ratings**  
(Groups of Four Identical Panels)

Differences	Occurrence Percent
0.5 .....	60
1.0 .....	35
1.5 .....	10
2.0 .....	5
2.5 .....	1

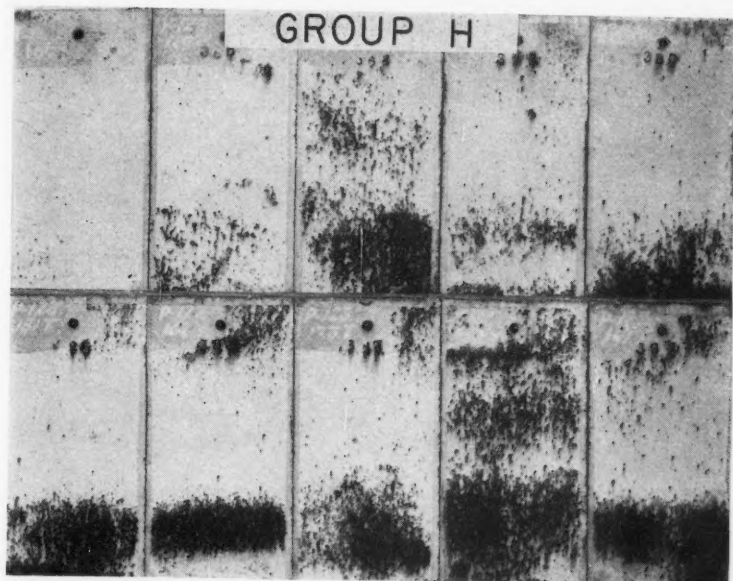


Figure 2—A second group of ten duplicate panels after three months' tide range exposure.

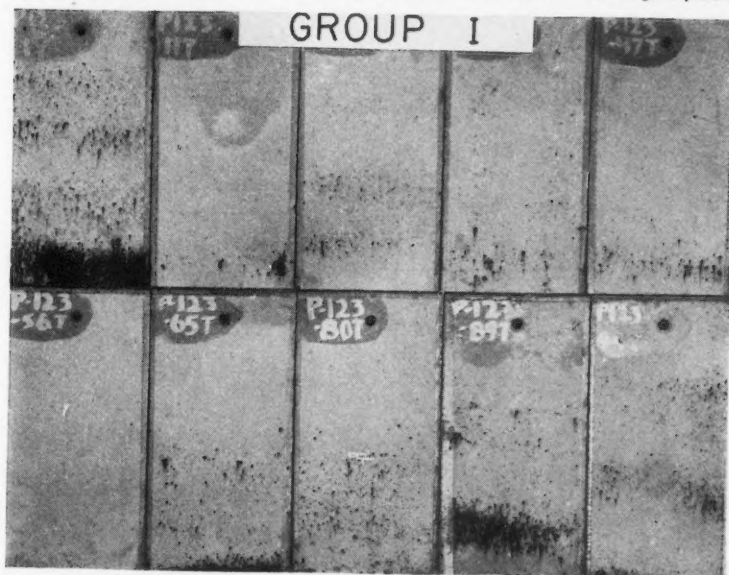


Figure 3—A third group of ten duplicate panels after three months' tide range exposure.

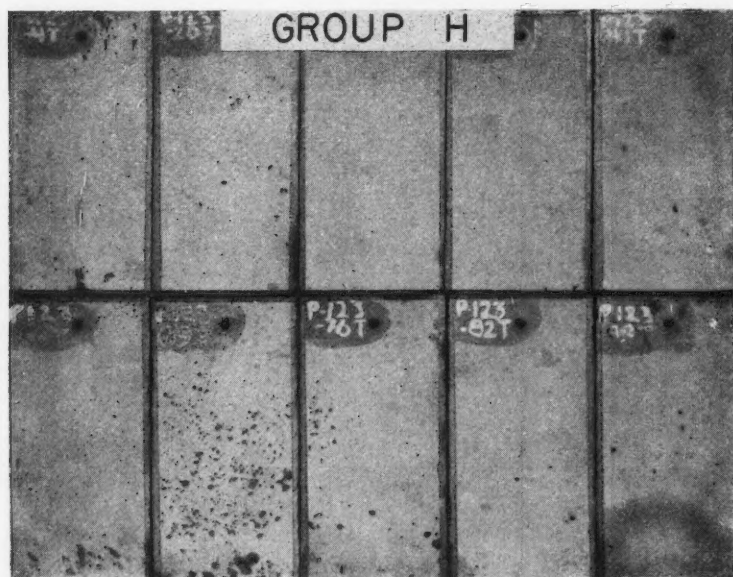


Figure 4—A fourth group of ten duplicate panels after three months' tide range exposure.

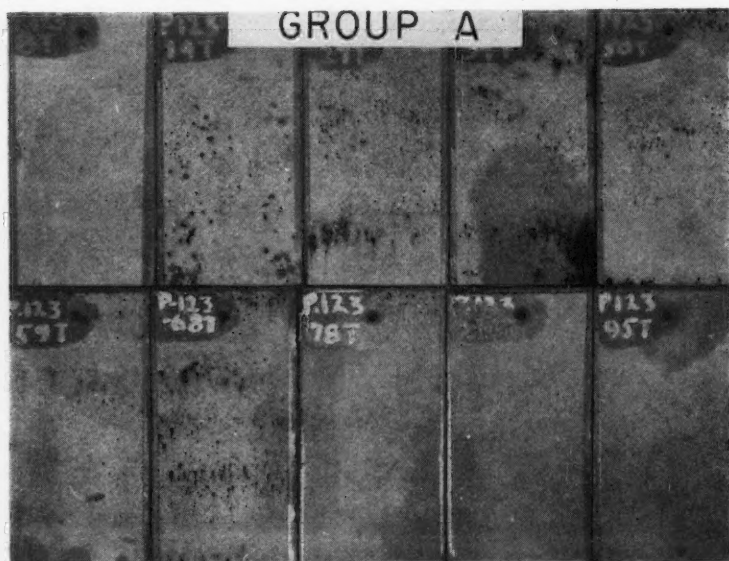


Figure 5—A fifth group of ten duplicate panels after three months' tide range exposure.

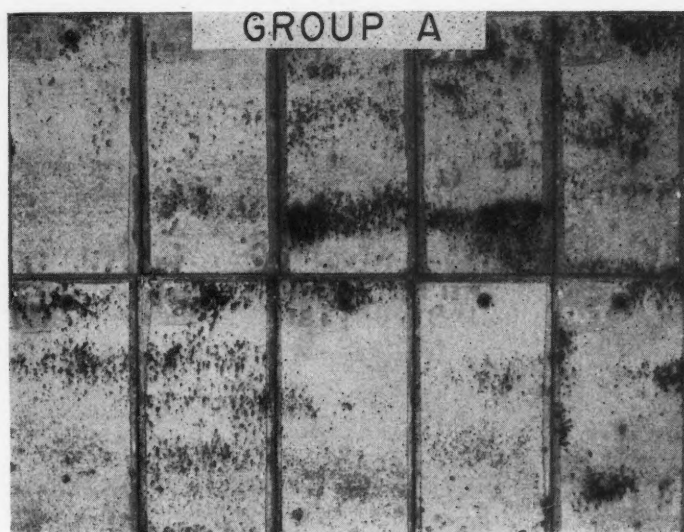


Figure 6—The same group of panels shown in Figure 5 after six months' tide range exposure.

exact prediction for future use. For example, suppose we were to expose two groups of four panels each, all panels and conditions being identical to any one of the groups previously discussed. Again, suppose this experiment to be repeated a large number of times. If we then consider the difference between the average rust ratings of each group of four panels, the probable occurrence of purely chance differences will be as shown in Table I.

Now assume that we perform the same experiment, but in this case the panels in each group differ in some particular from the panels in other groups. If the difference between the average rust rating of two groups were one unit, little, if any, significance could be attached thereto, since this difference might be due solely to chance one-third of the

time. If the difference were two units, we could conclude that a real difference existed. Of this, we are certain 95 percent of the time. This appears to be a satisfactory level for such work.

In statistical language, we may then say that the least significant difference between averages of four-panel groups, under the conditions of this specific test, is about 2.0 rust-rating units at the 95 percent probability level. For larger and smaller

TABLE II  
Least Significant Differences Between  
Average Rust Ratings

Averages of	Approximate LSD*
10.....	1.0
5.....	1.5
4.....	2.0
2.....	2.5
1.....	4.0

\*Least significant differences. 95 percent probability level.



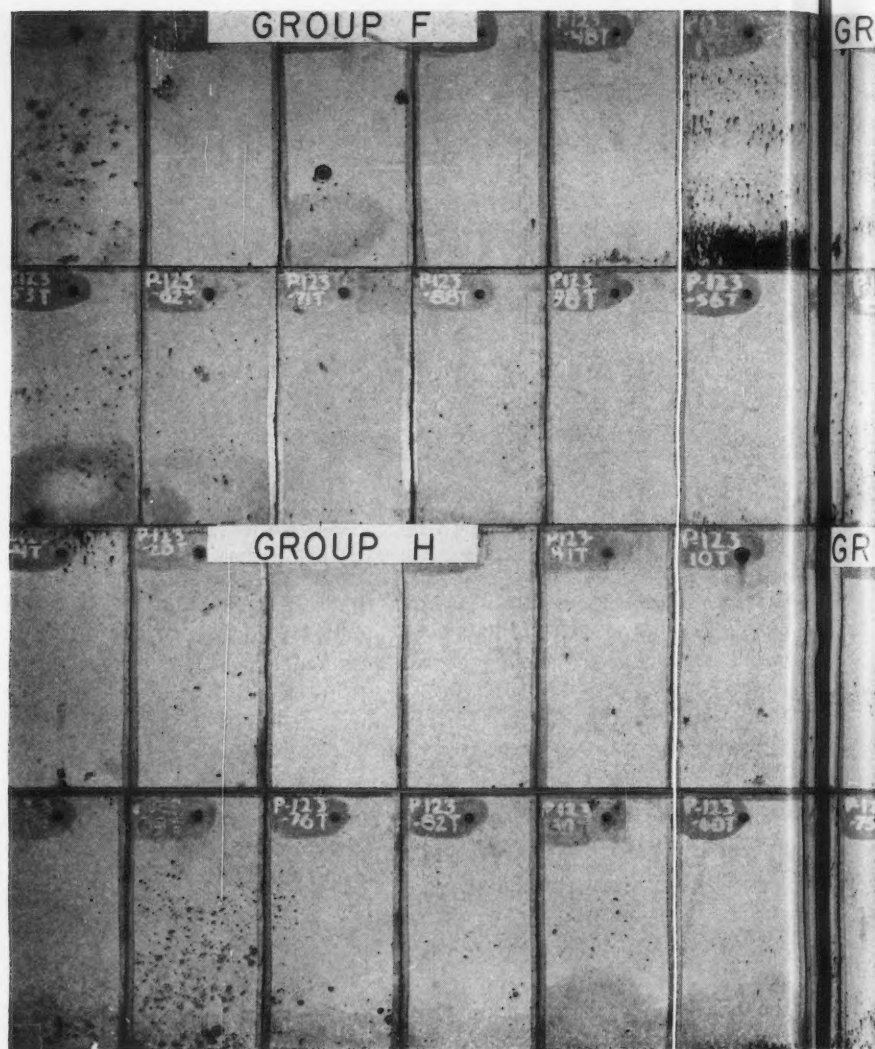


Figure 7—Four groups of panels arranged for comparison.

groups, again working at the 95 percent level, the least significant differences are as shown in Table II. Thus, the exposure of ten duplicate panels will serve to distinguish be-

tween groups even when the difference in average rust rating is only about one unit. While statistically significant, such differences may not be practically significant. While the



## GROUP I

## GROUP J

must be exposed to achieve discrimination at various levels.

It cannot be assumed that these figures apply under other conditions. The variation in tide range exposure results is obviously less serious when multiple-coat exposures are made as shown by Figures 8, 9 and 10, which have been selected at random from other exposure series.

It is regretted that we do not as yet have statistics on various types of exposures. It is hoped that this discussion has indicated not only how tide range exposures vary, but also how statistical studies may be of value both in the design of test and evaluation of the data.

The assistance of M. Kaplan of this laboratory, who supplied all statistical data, is gratefully acknowledged.

The opinions expressed are those of the author, and do not necessarily represent the opinion of the Navy Department.

T. R. DONLAN:<sup>2</sup> How did you clean the panels?

MR. CRANMER: We were trying to evaluate methods of surface preparation. Therefore, each group of ten panels was cleaned by a somewhat different method. There is no comparison of paints in the test since the same paint was applied to all the panels. The comparison is between methods of surface preparation and they involve such things as phosphoric acid treatments, dichromate and phosphoric acid, sulfuric acid, descaling, plus lime rinsing, etc.

E. J. DUNN, JR.:<sup>3</sup> Is there some set procedure that you followed in this statistical summary? I wonder how

question of practical significance must be determined by other considerations, it is obvious that data of this type will allow us to predict in advance exactly how many panels

<sup>2</sup>. Chemist in Charge, Solvents Development Lab., Standard Alcohol Co., Elizabeth, N. J.

<sup>3</sup>. National Lead Co., Brooklyn, N. Y.



Figure 8—A group of multiple coat panels after six months' tide range exposure. Vertical pairs are duplicates.

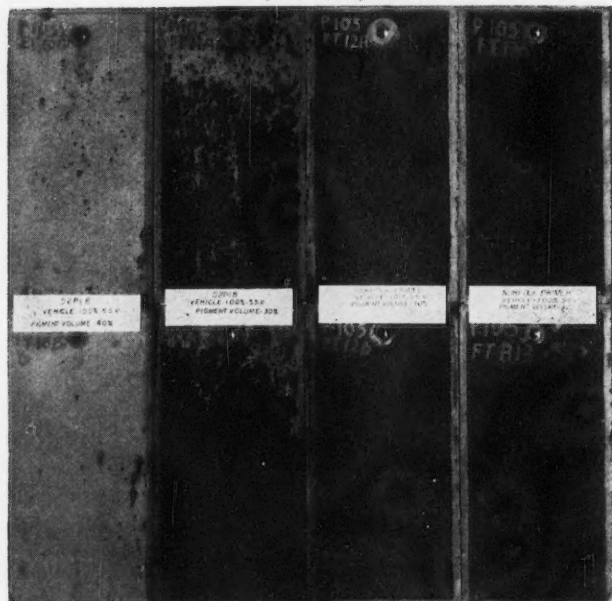
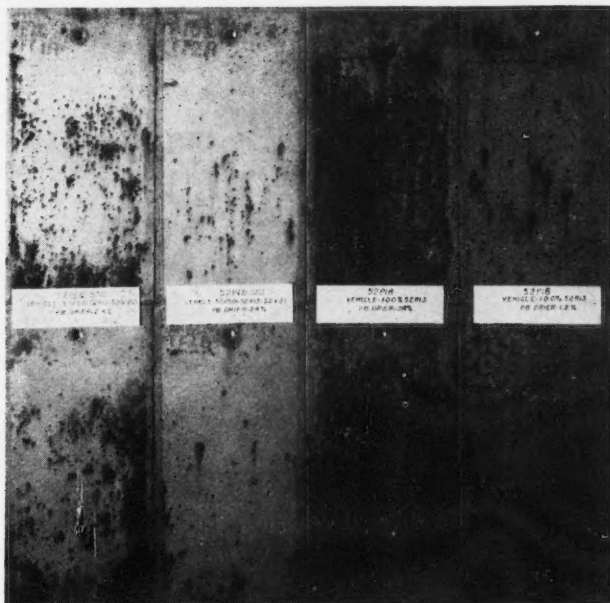


Figure 9—A second group of multiple coat panels after six months' tide range exposure. Vertical pairs are duplicates.

Figure 10—A third group of multiple coat panels after six months' tide range exposure. Vertical pairs are duplicates.



you could get so complete an analysis with so few panels.

MR. CRANMER: I was afraid somebody would ask a question like that. I am not a statistician, therefore I should suggest that any questions concerning the statistics be directed to M. S. Kaplan at our laboratory.

(Since the meeting, the following explanation has been received from Mr. Kaplan by the author.

(I assume that by statistical summary you refer to Tables I and II. It should be made clear in practice that such tables cannot be drawn up on the basis of a single exposure of 100 panels. To obtain a good measure of chance differences which might be expected between panel rust ratings under a given set of exposure conditions, it is necessary to expose many sets of panels in order to build up a reliable body of experi-

ence. The tables were prepared on the assumption that the uniformity or reproducibility obtained from 100 panels originally exposed would prove reliable. If this assumption is taken as true then the tables may be calculated by elementary statistical methods described in texts on the subject. No claim is made, however, that the values obtained in the particular experiment are reliable for prediction as to what might be expected. For interpreting the particular experiment, however, they can be used).

#### T. R. Donlan<sup>4</sup> (prepared discussion)

During the 1941 meeting of Subcommittee VII of Committee D-1, the question arose as to whether spot or partial sanding of steel test panels would adversely affect the life of the

<sup>4</sup> Chemist in Charge, Solvents Development Lab., Standard Alcohol Co., Elizabeth, N. J.

finished coat. The Army Ordnance wished to prepare specifications dealing with the reclaiming of used as well as rusted panels and the question, therefore, was brought forward at that time.

A coöperative test group was established at the February, 1944, meeting and the following program agreed upon:

(a) Naphtha degreasing versus vapor degreasing.

(b) Spot sanding versus full sanding, versus no sanding on degreased panels. Six panels were used for each test and all panels were cleaned in the same manner by the various coöperators. Each was assigned certain accelerated exposure tests depending upon available equipment. Accelerated tests consisted of:

(a) Distilled water immersion at 77° F.

(b) Tap water immersion.

(c) Humidity cabinet.

(d) Salt spray.

(e) Salt solution (20 percent NaCl) immersion.

Outdoor exposure tests also were run for check purposes.

The panels conformed to requirements of ASTM. Tentative Method for Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service (D 609-42T)<sup>5</sup> for cold-rolled steel panels. Spot and full sanding were carried out by sanding the lower half of each panel to a bright finish and spotting the upper half using a five spot pattern.

Panels were coated 1 mil thick with Ordnance Department Specification AXS-750 Primer and 1 mil with AXS-752 enamel. All but one coöperator edge-coated as supplementary protection.

The data showed variations too complicated to be covered in the present discussion. Attention, therefore, is directed to the official report by Subcommittee VII of Committee D-1, to be published in the current ASTM Proceedings.

Results, however, can be summarized as follows:

1) The six panels in each set, in general, checked quite well.

2) Spot sanding on the upper and full sanding on the lower part of the panel resulted in localized blistering and rusting not necessarily on the sanded areas. Failure, apparently, is governed by the type of accelerated exposure combined with the method of cleaning.

3) The presence or absence of supplementary edge-coating seemed to influence the location of rusting, that is, whether rusting occurred on the sanded or unsanded portion.

The importance of a uniformly treated surface on steel panels for immersion tests of paint systems was evident throughout.

#### V. M. Darsey<sup>6</sup> (prepared discussion)

Early work of the members on the Panel Preparation Group of Subcommittee VII reveals that, in addition to the cleanliness of steel, its surface finish influences the performance of applied paint coatings. Any method of preparation which changes the surface characteristics of the metal is likely to have a definite effect on the adhesion and resultant durability of applied paint coatings. For this reason a method of surface

5. 1944 Book of ASTM Standards, Part II, p. 1558.

6. President, Parker Rust-Proof Co., Detroit, Mich.

preparation which did not materially change the surface characteristics of the metal was recommended for cleaning steel panels prior to painting.

One of the major problems in specifying a suitable steel panel for paint tests is that of a uniform surface finish. Objection was raised against the use of full-finished, cold-rolled steel because of the poor adhesion of paint applied thereon. Originally it was suggested that a type of steel finish such as produced by sand-blasted rolls be used, which would provide better paint adhesion. Samples of steel panels secured from different sources with this type of finish were found to vary considerably in roughness, which was undesirable. In order to provide a more uniform steel finish, this problem was discussed with ASTM Committee A-1 on Steel, which committee advised that the No. 5 dull finish as produced by the Carnegie-Illinois Steel Corporation would be the most uniform type of surface commercially available. This type of steel finish is now recommended for use in preparing panels for paint testing by ASTM Tentative Method D 609.

**W. F. Singleton,<sup>7</sup>**  
(prepared discussion)

One important consideration in testing by humidity or immersion is the choice of a steel panel having a uniform surface of the requisite activity in regard to corrosion. In ASTM Tentative Method Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products

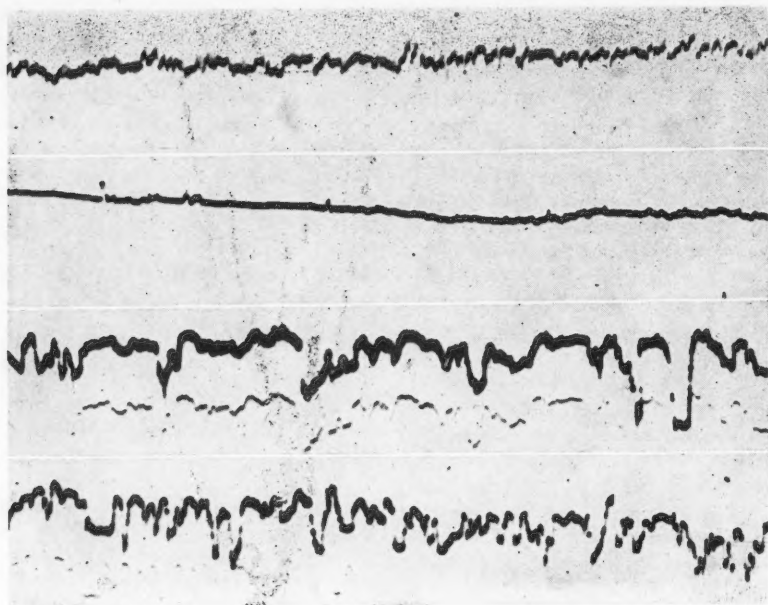
(D 609-45T)<sup>8</sup> there is a description of cold-rolled steel for panels which represented an agreement on a commercially available uniform surface finish. However, there had been less consideration given to the activity in regard to corrosion than to the physical roughness. In this connection, an experiment has been carried out on the effect of the surface condition of steel panels in immersion testing. The experiment concerns two variables, the roughness of the rolled surface and the practice of sanding the panels.

Specification D 609 now calls for a satin finish, equivalent to Carnegie-Illinois No. 5. Such a surface is visibly rough. For the experiment, a similar type of steel was on hand, except that the surface was slightly coarser. This may be called "rough" steel to distinguish it from a second type of steel, which may be described as "smooth." The latter, strip steel with No. 2 finish, is smooth enough to give a somewhat diffuse mirror reflection. It has parallel microscopic ridges formed by the machining marks on the finishing roll, as compared with the random peaks and hollows in the "rough" steel.

To characterize the surfaces more exactly, profilograph tracings were made (Figure 11). From the tracings, the average roughness in micro-inches from peak to valley and the frequency in waves per inch were calculated. The data are presented in Table III. Each surface was also rated according to the General Electric roughness standards, as shown in the last column of the table.

Each steel was prepared for painting by two methods. The first, corresponding to Procedure A of D 609, consisted of five operations:

<sup>7</sup> Philadelphia Lab., E. I. du Pont de Nemours and Co., Inc., Philadelphia, Pa.  
<sup>8</sup> 1945 Supplement to Book of ASTM Standards, Part II, p. 192.



**Figure 11—Surface roughness of cold-rolled steel.**

Original magnification: Vertical 5400 times, Horizontal 87 times. (Reduced one-half in reproduction).

Top section—Carnegie-Illinois No. 5 satin finish as specified in D-609.

Second section—"Rough" steel (see text).

Third section—No. 2 finish parallel to rolling direction.

Bottom section—No. 2 finish perpendicular to rolling direction.

(a) Vapor degreasing with trichlorethylene.

(b) Wiping with a cloth dampened with petroleum naphtha to remove the insoluble residue.

(c) Wiping with dry muslin.

(d) Immersion in isopropyl alcohol for  $\frac{1}{2}$  hour.

(e) Drying  $\frac{1}{2}$  hour at 150° F. The second method was by sanding.

(a) Vapor degreasing with trichlorethylene as before.

**TABLE III**  
**Roughness of Steel Panels**

PANEL	DESCRIPTION	PROFILOGRAPH		
		Average Roughness Peak to Valley, Microinches	Average Frequency, Waves Per Inch	General Electric Roughness Standard
Revision to ASTM D 609-45T...	Satin finish equivalent to Carnegie-Illinois No. 5.	98	270	D
Rough.....	Similar to revised D 609-45T, but coarser.....	124	104	E
Smooth.....	Cold-rolled strip steel No. 2 finish across machining marks.....	45	750	C to D
	With machining marks.....	17	....	....



(b) Sanding with No. 240 emery paper until the surface was uniform and all scratches and surface imperfections had been removed.

(c) Polishing with No. 400 emery paper to a mirror surface.

(d) Wiping with a clean cloth dampened with petroleum naphtha to remove the debris from the sanding operations.

All panels received a two-coat automotive paint system the same day they were cleaned, consisting of 0.5 mil of baking primer followed by 1.5 mils of a baked gloss topcoat. The panels were aged one week at room temperature. They were then tested by partial immersion in a bath of distilled water at 98 to 100° F. Each system was exposed in triplicate.

The results are presented in Table IV as the days to failure by blistering for each panel and as an average for each system. The reproducibility is good for most systems, and in all cases the difference between systems is larger than any panel variation.

The effect of surface condition on the rate of failure is found to be surprisingly large. The weakest system, on solvent cleaned rough steel, fails five times as fast as the systems on sanded steel. The data support two conclusions:

1) Sanding greatly alters the surface and delays the appearance of blistering. In this test, the sanded rough steel produced somewhat slower blistering than the sanded smooth steel.

2) For solvent cleaned steel, the rough surface produces faster blistering.

A tentative explanation of the effect of sanding is suggested by a further experiment, which is too in-

TABLE IV  
Effect of Surface Condition on Blistering

STEEL	Cleaning Method	Days to Failure	Remarks
Rough.....	Sanded	15+ 15+ 15+	Excellent at 15 days
Smooth.....	Sanded	15+ 15+ 15	
Smooth.....	Solvent cleaned	8 8 8	
Rough.....	Solvent cleaned	5 2 2	

complete to report in detail. An attempt to measure the surface activity by a modified Mears test indicates that the sanded surfaces are less corrodible than those which are solvent cleaned. This is the opposite of what had been expected on the assumption that sanding would insure a fresh surface of metal. Actually, it may be suggested that thorough sanding, as in the present experiment, may contribute to the formation of an oxide coating which is substantial enough to influence the rate of blistering of paint applied over it. Considerable mechanical work is done during the process of sanding, and the local heat which is developed may result in a heavier and more continuous coating of oxide than is present originally. The fact that the final sanding is carried out with a fine grade of emery paper would be more favorable to the formation of a continuous coating than ordinary procedure where a coarser grade is used. The superiority of the "rough" steel over the "smooth" steel after sanding on this hypothesis would be the result of the greater work which must be done to sand the original surface to a smooth finish.

It appears that the satin finish steel now specified in Method D 609 represents an active surface, both because the steel has a rough finish and because it is solvent cleaned without sanding. Results obtained from humidity or immersion tests on this steel should not be compared directly with previous results obtained on steel with a different surface finish.

**C. C. Hipkins<sup>9</sup>**  
(prepared discussion)

There are some who feel that the Pangborn test panels prescribed in ASTM Tentative Method D 609<sup>9</sup> are too rough, particularly for testing enamels. As a result, a compromise has been proposed to employ steel panels in accordance with ASTM Standard Specifications for Cold-Rolled Strip Steel (A 109-38)<sup>10</sup> having a No. 5 dull finish, as produced by the Carnegie-Illinois Steel Corp., or equal. There are others who feel that this proposed panel is too smooth a surface for paint testing purposes. Actually, the roughness is about that of standard C in the General Electric series, which has an equivalent roughness of approximately 16 microinches when measured by a profilometer and which, relative to a cold-rolled furniture steel, could be considered quite rough. In my opinion, the matter of whether a surface is considered too rough or too smooth is largely influenced by the type of surface one has been accustomed to employ for test purposes.

Let us examine the matter of just what is needed. The paint technologist must be concerned with the sur-

face condition of test specimens because of its relation to the adhesion of the finish to the substrate. The bond of a film to a surface is made up of two forces, one of which is mainly physical and depends upon the surface contours of the metal and the second is intrinsic adhesion, which is more physicochemical in nature and is a function of the attraction between the molecules of the metal and the molecules of the film.

As is well known, the mechanical roughening of the surface, such as by sand or grit blasting, provides a surface which in general insures a high order of adhesion. This treatment makes for uniformity of surface by removal of contamination, oxide coatings, etc. In addition, it also extends the actual area per unit of microscopic area so that there becomes a greater number of molecules available at the paint-panel interface to which the molecules of the film-forming materials may attach themselves. Further, blasting also provides many tiny facets of metal inclined at various angles into which the film can physically key itself. Profilometer measurements made at our laboratories employing a finely pointed diamond stylus for following the contours of a surface and an optical lever system for magnifying the undulations of the stylus as it rides over the surface under examination, lead to calculations that in some instances showed the increase in surface area to be at least 20 times. With changes of this magnitude in surface area, it is not at all surprising that blasting of surface increases adhesion to the extent that it does.

<sup>9</sup> Bell Telephone Labs., New York, N. Y.

<sup>10</sup> 1944 Book of ASTM Standards, Part I, p. 96.

While blasting the surface of a metal makes it uniform, care is necessary to maintain that uniformity, since it has been observed that it is merely a matter of minutes before the surface may change by oxidation and contamination from the atmosphere. This illustrates the desirability of minimizing the interval between the surface preparation and the coating if controlled uniformity of specimens is a factor in any particular set of tests.

From what has been said, it is at once apparent that the surface condition is an important adjunct of the finishing system, and it therefore follows that the only way to be certain that a particular film would adhere to a particular surface would be to follow the obvious method of testing that finish on that surface in a statistical manner. This is akin to the actual service approach to the problem of testing such as discussed by Mr. Elm.

However, where the properties of a protective coating are to be evaluated independent of the type of surface, or where the exact surface on which the finish is to be applied is not predictable, the more empirical type of approach to the problem can be followed by using an arbitrarily selected specimen on which to perform the tests. In these cases it really would not matter whether the Pangborn or the new proposed panel were used provided that either surface is uniform and readily duplicable and also that the results obtained are related to the surface on which the tests were performed.

I should like to caution against the hazard of expanding the results obtained in testing on one surface to predict the qualities of the same fin-

ish on a surface that may be considered but slightly different. To illustrate this point, some years ago we were startled by the wide differences in adhesion obtained on two brass surfaces. Our tests were conducted on rolled-brass sheet, which most of us would consider a smooth surface, and the adhesion of the finish was very good. However, when this finish was applied to brass parts made by a heading operation, its adhesion was surprisingly poor. Such a difference would not have been predictable on the basis of examination of the two surfaces since visually, at least, they were very similar.

Therefore, even when a finish has been found to possess a high order of adhesion to a given surface, it is advisable to check the adhesion on the particular surface to which the finish is to be applied, to insure satisfactory durability of the finish.

#### R. J. Wirshing,<sup>11</sup> (prepared discussion)

We take no exception to any of the statements made by Mr. Elm. We realize probably as well as anyone, that the preparation of test panels in the laboratory is a task that is difficult, time consuming, and requires considerable care if the results are to be of any value. This is due to the many variables, which include variations in the base metal, methods of cleaning, viscosity of the paint, temperature of the paint, method of application, thickness of film, type of undercoats used, baking times and temperatures, the amount of sanding or polishing of the topcoat, and probably others.

We agree with Mr. Elm that the preparation of the sample panel is

<sup>11</sup> General Motors Research Lab., Detroit, Mich.

of great importance, but we feel that it would be a physical impossibility to check each panel in any manner so as to be able to guarantee that each spot on the panel is the same as every other spot on the panel. While some chemical tests might be devised to insure this fact, the surface of the panel would thereby be changed and would no longer be satisfactory for the test. It is equally impossible to insure that panels in any series are uniform in all respects.

We are in the fortunate position of having very complete service data on a large number of paint materials since we have millions of cars in service under a wide variety of climate conditions. For this reason we can always have a standard material with which to compare the sample under investigation. In every case, such a standard is included in the test.

Panels are prepared for test in the same manner in which the part is prepared in production. For example, if the material is to be used on phosphate-coated steel, the panels are given a phosphate coating; if the material is to be used over a dip-coated primer, the panels are dip-coated in the primer; if the material is to be used over an alkali-cleaned steel, the panels are alkali cleaned. As a matter of fact, in many cases the test panels are run through the production line, to insure that they receive the same treatment the metal will receive in production.

Mr. Elm mentions coating the edges of panels with some protective coating such as wax. In automotive production, rusting from the edges is critical, so tests with large edge effects are most interesting and

we do not protect the edges of test panels.

The use of the immersion test instead of the humidity test would probably be satisfactory, but would be somewhat more difficult for us since we subject production pieces, including fenders and entire automobile bodies, to the same tests we use on panels.

We try to minimize the effect of some of the variables by preparing several panels of each material and by repeating the whole series several times.

While we are not sure that Mr. Elm is referring to the type of test work we are doing, nevertheless, we do feel that he is proposing an ideal set of conditions, which it would be impossible for us to obtain in our work.

**M. Van Loon<sup>12</sup>**  
(prepared discussion)

Mr. Elm has pointed out that to avoid misleading results in laboratory test procedures the panels used in the test must be uniform. This means that the ideal situation calls for uniformity not only over the entire surface of each panel, but also uniformity panel to panel throughout the entire group in the test. It becomes obvious immediately that we must not only specify the nature of the steel panels, but we must make certain that prior to the application of the coatings under test, the panel surfaces are uniformly free from oil, grease, rust and foreign matter. Method D 609 calls for a new prime cold-rolled steel that has been oiled at the mill, and therefore methods of removing this oily coating must be considered.

<sup>12</sup> Director of Paint Research, Sherwin-Williams Co., Chicago, Ill.

Usually the oily coating on such cold-rolled steel is of the paraffin-base type, sometimes with lanolin, fatty acids, sulfonated compounds or other wetting agents added. There will always be some dirt or other foreign matter present in the oily film. Whatever coating there is must be removed completely so that a uniform metal surface is obtained, such as is usually defined as "chemically clean." Recommendations have been set up for metal cleaning requiring that the degree of cleanliness be determined by the ability of the surface to support a film of water break-free.

When methods of cleaning the metal surface were considered, alkali cleaning, a very popular commercial method, was not included in the test procedure. Considering the very wide variety of such cleaners which might be used, there may be some difficulty in accomplishing the removal of all traces of residual alkali.

To prevent any chemical change in the character of the steel surface, only volatile solvents which are unreactive with the metal surface are used. The procedure for cleaning test panels in a suggested revision of Tentative Method D 609-45 T recommends the use of a grease solvent consisting of a mixture of three parts by volume V.M. and P. naphtha and one part monoethyl ether of ethylene glycol. It is reported that this combination has been found most satisfactory for removing oxidized films and foreign matter. It was originally intended that the method call for cleaning by scrubbing with a brush or lint- and size-free cloth followed by wiping dry with a clean cloth. The cleaning proceeded through the use of this sol-

vent mixture, without resorting to sanding or abrasion of the surface, until the panel would support a water film break-free when immersed in distilled water.

This portion of the method deserves further discussion. At least one possible interpretation of the method in its original form was that the cleaning may and can be done entirely by solvent wiping to the point where the surface will support a break-free water film. Since the oil or other surface soil on the panels may vary widely in character and therefore, in ease of removal, it is possible to clean some panels by this method to the point where they will support a film of water break-free over substantially the entire surface of the panels. In the case of our own laboratory stock of panels, however, it has not been possible to do so without expenditure of time and effort which we consider excessive, particularly when a large number of panels are used in a given test. Furthermore, there is usually a wide variation in the degree of the uniformity of cleanliness and of the surface activity of the panels, which is contrary to our original concept of conditions required for laboratory test procedures.

It would appear that any solvent scrubbing or wiping as ordinarily conducted is immediately accompanied by some surface contamination. It is impractical to provide a continuous supply of clean cloth and clean solvent so that the contaminated cleaning cloth never travels back over the cleaned areas.

Vapor degreasing with trichlorod —or perchlorethylene would eliminate many of these objections, since if properly executed the metal sur-

face is cleaned with fresh pure condensed solvent. Sometimes it is found desirable to include some jet cleaning to remove insoluble dirt particles which cannot be removed without some mechanical force. We have found that panels degreased with trichlorethylene vapor will support a film of water break-free in spite of the obvious presence of mechanical dirt or smut. This deposit should be wiped off, however, if the panel is to be used for laboratory tests.

Usually, in commercial practice, if the shape of the article being cleaned is such that proper washing action is obtainable, solvent vapor degreasing is very satisfactory. However, because laboratory scale apparatus is not always available for such cleaning, because some experience and care is required in handling solvent, and because of some apprehension as to possible activation of the metal surface, it was originally decided not to suggest trichlorethylene or perchlorethylene vapor degreasing as an alternate cleaning method for preparing steel panels for the laboratory testing of organic coatings. It is now being considered as an alternate method of cleaning to be included in the recommended method.

A proposed revision of Method D 609 that has been under discussion states, that after the initial solvent scrubbing, "The panels shall then be further cleaned . . . by the use of this solvent mixture until each panel cleaned will support a water film break-free when immersed in distilled water." It will be noted that this did not restrict the solvent cleaning to scrubbing only.

In an attempt to improve on solvent wiping to clean laboratory steel test panels, we use in our laborato-

ries what we call the solvent spray cleaning method. This method is simple, fast and economical. It requires no special apparatus beyond a regular spray gun, and the solvents used are readily available in the average paint or varnish laboratory. A large number of panels can be brought to a state of uniform and satisfactory cleanliness where they will support a film of water break-free. Fresh solvent is all that is used at the point of cleaning, and the advantages of mechanical action or jet cleaning are inherent in this method.

The panels are used as they are taken from the stock bundles with no preliminary removal of the mill oil coating. A sample immersion in distilled water will serve to check whether sufficient hydrophilic surface-active agents are present to support a film of water break-free, in spite of the presence of oily contaminants. If so, the water-soluble agents are removed by running water over

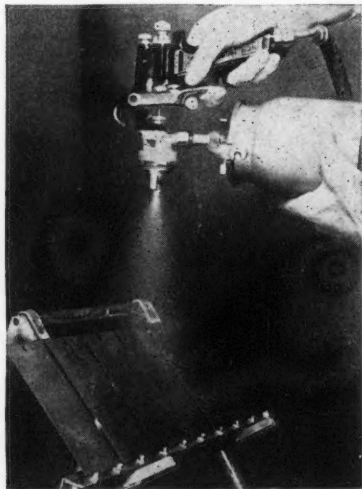


Figure 12—Method of solvent spray cleaning.



the panels until they will not support a break-free film of water. This will assist in establishing the validity of the final water film test.

Figure 12 illustrates the proper method of solvent spray cleaning. The test panels with their shop coat of oil and incidental soil are mounted on a spray rack as shown. The nature of some rust inhibitive oils used on stock panels indicated that the degreasing solvent should be a mixture of aliphatic and aromatic types. We usually use a mixture of half V.M. and P. naphtha and half xylene, but we have found the blend of three parts naphtha and one of cellosolve about equally efficient. There probably are others equally good.

The solvent mixture is sprayed from a conventional spray gun operated with the fluid tip wide open, but with the atomizing jets stopped down almost completely. This concentrated the spray in a much smaller area than is ordinarily used in painting.

An important factor in the method is the angle at which the spray is directed onto the panels. It should be projected from above at an acute angle to the panel (25 degrees or less) so that the washings are rinsed off ahead of the spray and not forced around to the other face of the panel. If the solvent spray strikes too nearly perpendicularly to the face of the panel, the washings are forced around the edges onto the other side of the panel. The spray is swept back and forth several times across the panels, starting at the top and working down as the soil is washed ahead of the spray. A pan is placed below the panels to catch the washings.

The technique used is exactly the same as one employs in cleaning sand

and leaves off a sidewalk using a garden hose. The oil and dirt are driven off the panels with a coarse spray of solvent so that only clean solvent strikes at the point of cleaning or over the area already washed.

The backs of the panels are usually degreased first, then the face sides, after which they are allowed to dry. After the panels have been cleaned, the usual precautions should be taken to avoid contaminating the panel faces in handling. When they are dry they are polished by rubbing briskly with a clean, dry cloth. This last operation removes the finely divided dirt which was originally bound to the panel by the oily film, and furthermore seems to be a necessary step in conditioning the now oil-free surface so that it will support a film of water break-free. The beneficial effects of polishing are apparent with almost any method of cleaning.

For routine laboratory tests, which may involve a relatively large number of panels in a given set, the normal practice is simply to allow the solvent spray cleaned and polished panel to air dry briefly and then proceed with the application of test paints. However, for precise testing, the method would require the testing of each panel by immersing it in distilled water to determine whether it will support a film of water break-free. The panels should then be immersed in alcohol for 10 minutes, followed by air drying or force drying at 125 to 150° F. for 10 minutes.

The 10-minutes immersion in alcohol is beneficial where there have been any deficiencies in cleaning up to that point. Where panels were solvent cleaned by wiping, the portions of the panel surfaces which

would support a film of water break-free rose from an average of approximately 15 percent to 80 percent in one instance, from 45 percent to 85 percent in another.

When the panels are force dried for 10 minutes at 150° F., in some instances there is a drop in the water break rating, which may be due to deep-seated contaminant which has not been entirely eliminated in cleaning. Trichlorethylene vapor degreasing is thorough, so that the effects of either alcohol immersion or force drying are not very noticeable. The same is true to almost the same extent in solvent spray cleaning. Where cleaning is less efficient as in the case of solvent wiping, the effects may be more pronounced.

The comparisons are interesting when viewed side by side.

The first column of Table V gives the method of cleaning and the solvents used. The next two columns give the percentage of the surface area for the two faces of each panel which would support a film of water break-free; first, after cleaning to the point where the water-break test is called for in the specification, and,

second, after 10-minutes immersion in alcohol and air drying or force drying as indicated. All the figures are averages taken from six panels in each group.

The next four columns present the results of a modified Mears activity test, using a two percent hydrogen peroxide plus 0.006 percent sodium chloride solution, twelve spot tests on one face of each panel.

Thus the "over-all average" represents the average of 72 spot tests for each method.

It will be noted that ratings on the water-break test are rather low and vary rather widely using solvent wiping. The blend of V.M. and P. naphtha plus xylene is somewhat more efficient than V.M. and P. naphtha plus cellosolve. The 10 minutes soak in alcohol brings up the water-break rating of both, but the drop on force drying is greater with the blend using cellosolve than that using xylene.

Solvent spray cleaning and trichlorethylene vapor degreasing get high ratings on the water-break test. In this instance, the blend using xylene shows a greater drop on force drying

**TABLE V**  
**Panel Preparation, Water-Break Tests, and Activity Rating Averages**

Panel Preparation	WATER-BREAK TEST, PERCENT		Modified Mears Activity Tests, Sec.			
			Average Maximum	Average Minimum	Over-all Average	Difference—Average Maximum and Minimum
	Initial	Final				
Solvent wipe V.M. and P. naphtha + cellosolve...	12 and 19	82 and 78	36	15	24.5	21
Solvent wipe and bake V.M. and P. naphtha + cellosolve.	34 and 30	39 and 54	29	10	20.7	19
Solvent wipe V.M. and P. naphtha + xylene.	47 and 68	82 and 83	35	11	23.5	24
Solvent wipe and bake V.M. and P. naphtha + xylene.	45 and 60	80 and 86	28	12	19.4	16
Solvent spray V. M. and P. naphtha + cellosolve	99 and 98	99 and 97	21	12	15.5	9
Solvent spray and bake V. M. and P. naphtha + cellosolve	100 and 98	98 and 98	27	12	18.2	15
Solvent spray V. M. and P. naphtha + xylene.	100 and 99	98 and 98	18	10	13.6	8
Solvent spray and bake V. M. and P. naphtha + xylene.	100 and 100	82 and 82	29	16	23.0	13
T. C. E. degrease.	100 and 100	100 and 100	48	17	26.4	31
T. C. E. degrease and bake.	100 and 100	98 and 100	39	14	23.9	25

than with the cellosolve blend. It may be worth noting that while the trichlorethylene degreased panels show no loss in water-break rating after force drying, this force drying was done at 150° F., whereas trichlorethylene boils at about 190° F. In effect, the trichlorethylene panels already have been subjected to higher temperatures before the original water-break test than they encounter later.

Turning to the activity tests, the solvent wiped panels show rather wide differences between the average maximum and minimum activity ratings. The solvent spray cleaned panels show the highest degree of activity and the best uniformity of activity. It is the only one to show a decrease in activity after force drying, whereas the others rather surprisingly show an increase in activity on baking even though the water-break rating dropped. In spite of the high water-break rating of the trichlorethylene vapor degreased panels, they show a relatively low degree of activity and the widest differences in activity.

#### Solvent Spray Cleaning Method Apparently Most Satisfactory

Summing up, it appears that the solvent spray cleaning method shows considerable promise as a reasonably rapid and economical method of preparing panels for laboratory tests. We should remind ourselves of our original premise, which was to prepare a group of panels satisfactorily for laboratory test purposes; each panel uniformly clean, uniformly active over its entire surface, and the whole group uniform panel to panel. In this way we may be sure that any differences observed in the perform-

ance of the coatings on test do not arise from differences in the substrate. Solvent spray cleaning has come sufficiently close to this ideal to warrant further consideration.

#### I. C. Clare,<sup>13</sup> (prepared discussion)

At the Hercules Powder Co. Experiment Station, we have arrived at a series of routine procedures for preparation of panels. These procedures will not necessarily fit the needs of other investigators but they are suited to our requirements from the standpoint both of consistency of results obtained and the rapidity with which tests may be made.

Our work at Hercules on protective coatings may be divided into two major categories: first, the evaluation of oleoresinous finishes, both clear and pigmented, which dry by oxidation and polerization; and, second, the evaluation of lacquer and lacquer enamel coatings, which dry primarily by evaporation of solvent. Our objective in the first case is to determine the performance characteristics of resinous compositions for the purpose of resin development and customer information. In the second, we wish to determine the effect of various commercially obtainable resins and plasticizers on both established and experimental film-forming materials. Naturally, the two objectives frequently overlap. To obtain this information we have established a number of routine tests which involve both free and applied films. Insofar as is possible, ASTM procedures are used when available. In some cases, however, deviations from such procedures are found necessary.

13. Hercules Experiment Station, Hercules Powder Co., Wilmington, Del.

In our routine tests, the general appearance of the applied film, particularly gloss, flow out, depth, and fullness are determined. Drying rate, resistance to water, alkali, impact, abrasion, and discoloration when exposed to baking conditions and ultraviolet light, effect of gas combustion fumes, flexibility, water spotting, sweat-box or tropical test performance, cold-check, and general exterior durability of the applied films are studied. For a number of these determinations, glass, wood, and tinplate panels are employed. For other tests, however, it is necessary to use steel panels and it is with the preparation of these steel panels for use in such tests that we are now concerned.

In the testing of oleoresinous coatings, steel panels are used in determining flexibility, adhesion, impact, abrasion resistance, and durability. Primed steel panels are used for obtaining discoloration data on white and pastel shades of enamels. Steel panels are also used in determination of alkali, acid, and salt spray resistance, although occasionally the alkali tests are made on glass, using the common test-tube procedure. In the evaluation of lacquer coatings, steel panels are used in testing sanding properties, print and corrosive reagent resistance, sweat-box or tropical test performance, and durability. Occasionally, steel panels are used in determining cold-check resistance; although with furniture lacquers, birch, mahogany, or walnut plywood panels are more commonly employed.

Our steel panels are purchased in  $2\frac{1}{2}$  by  $5\frac{1}{2}$  and 6 by 12-inch sizes. They consist of oiled soft cold-rolled steel. A number of methods have

been evaluated for the preparation of these panels for coating. This has varied from solvent cleaning to sand blasting. One objection to the use of solvent cleaning is that the panels frequently are slightly rusted and sometimes have a somewhat rough surface which is developed during the rolling operation. They also have sharp edges caused by the method of cutting the panels.

The procedure currently used is a result of a number of experimental efforts, and for our purposes it appears to be quite satisfactory. The rough edges of the panels are smoothed off by filing. The panels are then cleaned in toluene or, if this solvent is not available, xylene. One face of the panel is then sanded until smooth, using a portable belt sander equipped with a No. 120 sanding belt, the original surface being entirely removed during the sanding operation. Sometimes, but not always, the sanded panel is polished with either 360 or 400 Wet-or-Dry Tri-Mite paper. This second operation is desirable but not essential and is done when time permits. During the entire sanding operation, the operator has been instructed to carefully refrain from touching the surface of the panels with his hands or fingertips. This may be avoided either by the use of gloves or by very careful handling. The panels are stored in toluene if they are not to be coated immediately.

It is recognized that the sanding operation accentuates the tendency of the steel to corrode under the film, particularly when subjected to tropical or exposure tests. However, we are primarily interested in the comparative performance of the coatings in a series. In a series of this sort

one variable has been changed systematically, and standard formulations of well-known performance characteristics are included for reference. Since each series contains these comparison standards, we are able to give the panel a phosphate or similar chemical treatment to passivise the panel surface in test where corrosion of the metal may cause complications.

By subjecting each coating system to identical tests, we believe that the effect of systematic variation of the ingredients under test are adequately brought out. Naturally, the panel surface cannot be treated when the panel is subsequently to be subjected to mandrel or other flexibility tests, but surface treatment is frequently used where we are interested in tropical or sweat-box performance and exterior durability.

Rust-preventive measures are always taken when the coating is formulated for an application where such measures are common commercial practice. Examples are automotive and refrigerator finishes. Naturally, if the coating is designed to have an inhibitive effect on corrosion, this chemical treatment of the panel surface cannot be carried out. In a case of this sort, we consider the sanded panels to give a more severe test.

We do not consider the water-break test a suitable standard for indicating the uniformity of this sanding operation, but have no suggestion for a more adequate method. A panel treated with a known amount of soil will show a water break when submitted to this test. If the soil is removed with suitable solvent, the panel will then pass a

water-break test, particularly if subjected to a mild sanding operation directly after cleaning. However, if the panel is sanded thoroughly and then polished so that the surface presented is as clean as we know how to make it, we find that the surface wets very poorly with water and a pronounced break is observed.

A study of the degree of cleanliness of such sanded and polished surfaces has been made by means of copper electroplating tests. For these electroplating tests, a rather simple apparatus was set up. The following materials are required:

- 1.5-, 2- or 6-volt battery, with necessary clips and wires.
- Steel panels, 25 by 50 mm.
- 150-ml. beakers.
- Copper anode 25 by 50 mm.
- 100-ohm resistance coil.
- Rochelle salts.
- Copper sulfate.

Using one cell of an ordinary 6-volt battery, the copper anode is attached to the positive pole. The panel under test constitutes the cathode and is connected to the negative pole through a 100-ohm resistance coil. The cathode and anode are suspended in the plating bath contained in a 150-ml. beaker by means of alligator clips supported by burette clamps attached to separate ring stands. The circuit is opened and closed by simply raising or lowering the cathode into the plating bath. With a distance of 45 mm. between anode and cathode, the current drawn was approximately 0.070 ampere (current density 0.0056 ampere per square centimeter).

Two solutions were used in the preparation of the copper-plating bath. The first contained 3 g. of hy-

drated copper sulfate dissolved in 50 ml. of distilled water. The second contained 6 g. of Rochelle salts in 50 ml. of water. When dissolved, the two solutions were mixed and diluted to 125 ml.

Referring again to the soiled, partially cleaned, and thoroughly cleaned panels, this last being polished after real dressing down of the surface with abrasive, we find that if the first two panels are subjected to a copper-plating operation using the described equipment, the soiled and partially cleaned panels show either no deposition or an irregular deposition of copper on the panel surface. The carefully sanded panel, however, plates smoothly and evenly, indicating, in our opinion, the thoroughness of the cleaning operation.

The water-break test has been used on a number of panels cleaned with different solvents and subjected to various degrees of sanding. It does not appear to give an adequate story since the more thorough the cleaning operation, the greater tendency there is for a water break to be observed. This water break is also observed on the soiled or unclean panels, but those which are intermediate in cleanliness, as determined by our electroplating tests, do not show the water break. Harris<sup>14</sup> has discussed the effect of anionic and cationic agents when adsorbed on metal surfaces. We believe that the wetting effect is obtained when a hydrophilic film is present on the metal surface. When this is removed entirely, the water break is no longer observed.

14. Harris, J. C., Metal Cleaning: II—Soil Removal Performance Methods. ASTM Bulletin 136, 31, Aug. (1945).

We do not recommend the electroplating method as a means of determining the suitability of steel panels for use in protective coating evaluations, although some systematic scheme for determining the efficiency of the cleaning operation might be developed by the use of this electroplating technique. This procedure was developed in our laboratory by B. S. Van Zile for the purpose of observing the efficiency of metal-cleaning compositions, and at a later date, will be described in more detail by Van Zile pursuing the original objective.

We hope that this discussion will not serve to confuse the problem, as we are presenting it for what it may be worth to assist in consideration and development of an adequate specification.

#### H. A. Nelson,<sup>15</sup> (prepared discussion)

It should be kept in mind that insofar as Tentative Method D 609 is concerned, ASTM is primarily interested in preparing panels of limited size, which are to be used for laboratory tests of the accelerated type; that is, immersion, humidity, salt spray, light and water exposure, etc. Therefore, this method must go beyond the requirements of average commercial surface preparation procedures in an effort to reduce the number of variables in these tests. We should, if possible, know what the cleaning procedure does to the uniformity of the surface of the steel panel, which should be as uniform as the uniformity of the steel itself will permit and no less. For this reason we are interested in tests to show that the cleaning procedure

15. Technical Department, The New Jersey Zinc Co., (of Pa.) Palmerton, Pa.



TABLE VI  
Experimental Results

	SURFACE UNIFORMITY				General Surface Activity, Mean of all Readings, Seconds
	Highest Reading, Seconds	Lowest Reading, Seconds	Mean of Higher Readings, Seconds	Mean of Lower Readings, Seconds	
Procedure which included a rinse in a reactive inhibiting agent (3 panels* with 16 spots on each panel).....	735+†	95	350	169	227‡
Solvent cleaning procedure (4 panels* 16 spots).....	125	20	100	55	75
Electrolytic cleaning as used in metal plating operations (4 panels* 16 spots).....	35	15	30	20	25

\* Panels about  $3\frac{1}{2}$  by 6 in. and of steel meeting the requirements of Tentative Method D 609.

† Next highest readings were 480+ on the second panel and 360 on the third panel.

‡ High reading indicates low order of surface activity.

has really cleaned the surface and not added to the nonuniformity by leaving localized contamination, or areas that are less subject to corrosion due to some reaction with the chemicals that are used. We should also like to know something about the general activity of the surface (or its tendency to corrode), but this is really secondary to uniformity and, of course, to reproducibility of the results obtained by the cleaning procedure.

When a panel passed the water-break test, we have, it is true, assumed that it indicates that the surface is as uniform as the characteristics of the steel permit. The fact that a break-free film of water may only indicate that hydrophobic matter is absent is disturbing, but it is the test with which we have had most experience so it cannot be discarded in an offhand manner. However, there has been some debate as to the place of the water-break test in the specification method. Some consider it a periodic check on the efficacy of the cleaning procedure, to be used only on occasional panels. Others think that it should be the final step on every panel, with the idea that contact with water has a stabilizing effect on the surface. Still

others prefer an alcohol dip for stabilizing and drying the surface and, as Mr. Van Loo points out, there is evidence in favor of this.

A test for exploring the effectiveness of cleaning procedures, as to producing uniformity and activity, is badly needed. The copper-plating test suggested by Mr. Clare should be studied. Another test, already mentioned by Messrs. Elm, Singleton and Van Loo and which undoubtedly has possibilities, is the spot corrosion test with dilute hydrogen peroxide solution. This was suggested by Mr. Reinhard of our laboratory as a simplification of the test used by Mears and Evans<sup>16</sup> in their study of the probability of corrosion on steel surfaces.

There is increasing evidence that by the application of statistical methods this simplified test can supply information about the degree of activity of the surface (tendency to rust) as well as the uniformity of this tendency, and that it should be useful for rating cleaning procedures. Experiments in our laboratory indicate that cleaning procedures may vary to a considerable degree in the time (seconds) re-

<sup>16</sup> Mears, R. B., and Evans, U. R., The Probability of Corrosion, Trans. Faraday Soc., 31, 527 (1935).

quired to produce first visible evidence of corrosion under a drop of hydrogen peroxide solution. Examples of such experimental results are shown in Table VI.

The solution used consists of 16 ml. of Superoxol (30 percent  $H_2O_2$ ) in 984 ml. of distilled water. Drops (one or two) from an ordinary medicine dropper are uniformly distributed in groups of four spots across and four uniformly spaced rows extending to the bottom of the panel. Each row of four spots is timed as a unit and, in order to average the starting time, the stop watch can be started after the second spots. This is permissible, because readings are recorded only to the nearest 5 seconds. The observer watches for the first indication of brown color in any of the small bubbles which form on the steel surface under the spot. The lighting should be adjusted to avoid reflections of the light source; fluorescent lighting can be used to advantage. Hydrogen peroxide solutions are sensitive and at times erratic and should be checked against standard panels periodically prepared and kept in a desiccator. (Some make the solution more active by adding NaCl, but the value of this is questioned.) Panels cleaned by the third procedure in Table VI are surprisingly reproducible and have been used as standards for checking the activity of the solution. This cleaning procedure may also prove to be useful as a relative standard for comparing the effectiveness of other cleaning procedures.

The results with the first procedure are interesting because of the indication that an inhibiting rinse may not react uniformly on steel surfaces unless the procedure is

more carefully controlled than it was in this case. So far, the results do not justify any conclusions as to possible relationships between surface uniformity indicated by a break-free water film and uniformity of readings obtained by the spot test.

### R. I. Wray,<sup>17</sup> (prepared discussion)

Mr. Elm presents a very interesting discussion of an important subject. The need for accelerated tests to evaluate paint coatings is indeed great, since many of the coatings now available do not have as much background upon which to evaluate them as some of the older types of coatings. Mr. Elm's analysis of the problem is very good and the various steps of procedure he proposes appear quite logical.

In preparing panels for painting, the necessity for eliminating sharp edges and corners, which might cause rapid failure of the paint, cannot be overemphasized. Their effect can be minimized by carefully rounding all edges and corners by filing. The use of fairly heavy gage metal (No. 14 to 16 gage) for the panels aids in permitting better rounding of the edges. The use of additional paint or wax coatings on the edges still further improves this condition, but it is difficult to paint a sharp edge satisfactorily.

In the application of the coating, the use of automatic spraying or dip-coating at a slow uniform rate will assure a more uniform paint film over the entire panel. This, however, does not eliminate the desirability of making accurate film thickness measurements.

<sup>17</sup> Aluminum Company of America, New Kensington, Pa.

The effect of scratches in the paint film is very important, as pointed out by Mr. Elm. If the paint films are to be deliberately scratched, the scratches should be small and uniform. Care should be taken that the scratches do not deeply score the metal surface itself. The practice of scratching paint films on metal panels is open to question for a number of reasons. This is particularly true if the metal has been given a special treatment, such as bonderizing, prior to painting, since the scratch will disrupt the protective film on the metal itself. As pointed out by Mr. Elm, it is important that differences in rusting be caused by differences in the protective coatings and not by differences in the corrosivity of the metal.

Our observations are in agreement with Mr. Elm's statement that more paint blistering occurs with the same paint system in fresh water than in salt water. His explanation appears to be logical. Blistering of the paint itself should not be confused with corrosion blistering as Elm points out. Results have been observed on aluminum panels similar to those on steel described by Elm.

In discussing the relative merits of testing by complete immersion in water and testing by exposure to high humidity, Elm reaches the conclusion that there is no difference in the amount of water absorbed by identical paint films exposed to the two environments. Several years ago we conducted a few tests on free paint films (not attached to any substrate). The paint film used in these tests consisted of one coat of aluminum paint made with a long oil varnish vehicle. It was found that

different amounts of water were absorbed when the paint film was immersed in distilled water maintained at 80° F. and when exposed to 100 percent humidity at 80° F. In a 24-hour period, the film which was exposed to 100 percent humidity showed a gain of 17.4 mg. of moisture per gram of film, while the film which was immersed in distilled water absorbed only 14.7 mg. of moisture per gram of film. The moisture permeability of the paint film was also found to be greater when exposed to high humidity than when exposed to liquid water.<sup>18</sup> Whether these relationships would hold when the paint films were attached to a metal substrate was not determined.

The interpretation of the results of accelerated tests in terms of the service life of the paint coatings is perhaps the most important phase of the problem. It should be pointed out that immersion tests or humidity tests have the most significance when evaluating paint coatings for service conditions involving exposure to moisture. The effect of aging of the paint on its embrittlement is not taken into account in an accelerated test. It might be easily possible to have a paint which would give exceptionally good results in an accelerated test of short duration and yet give inadequate protection to a metal surface for long periods because of the rapid embrittlement of the film with age. Short oil varnish base enamels sometime behave in this manner. It would, therefore, be necessary to establish a correlation between accelerated and service tests for each and every type of coat-

18. Edwards, J., and Wray, R. I., Permeability of Paint Films to Moisture, *Ind. & Eng. Chem.*, **28**, 549-559, (1936).

ing. Before starting an accelerated test the exact nature of the coating must be known. It might, thus, be difficult to evaluate any new type of coating on which no service experience existed. For most coatings, however, the statistical method of evaluation proposed by Elm should give the desired information and, as he points out, it should prove well worth the trouble in carrying it out.

**W. H. Lutz<sup>19</sup> and E. J. Lewis,<sup>19</sup>  
(prepared discussion)**

Mr. Elm's discussion is complete and logical. We feel that he has performed a service in focusing attention on fundamentals rather than on controversial details.

We are in accord with Mr. Elm's remarks on the importance of panel uniformity, surface preparation and activation in all tests involving metal panels. We also share his views relative to small test panels. We have standardized on 6 by 12-in. panels in order to reduce edge and corner areas as to a small proportion of the testing area. Because of our own difficulties with small panels, we are frequently surprised to note that most test panels offered by suppliers are of relatively small size. Elm's evaluation of the water-break test for panel cleanness agrees with ours.

Our experience with molten wax for protecting panel edges has often been disappointing. When molten, the wax tends to creep up the panel face; when cool it becomes brittle and breaks away unless handled very gently. We prefer to use several coats of spar varnish, when time permits.

We agree with Elm that protec-

tion from corrosion depends on film continuity and that minute pinholes and imperfections exert a tremendous influence on failure. We have found the salt-spray test distinctly useful in showing up film irregularities and we do not entirely share Elm's skepticism of this test. In our opinion, it reproduces the failures encountered under actual conditions of corrosion more closely than do water-immersion tests. In the salt-spray test, the metal corrodes but the paint film does not usually blister. If blistering does occur, it is purely a secondary effect. In immersion testing, blistering seems to be the predominant form of film failure.

Elm expresses surprise at the lack of agreement among experienced paint-testing engineers. Experts always seem to disagree whether they be paint test engineers, surgeons, or lawyers. Disagreement is a healthy sign, for it indicates that people are thinking. Most disagreement stems from honest differences of opinion. Opinions differ because there is insufficient factual evidence to support one sweeping conclusion. We need more facts in order to eliminate some of the controversial opinions.

One reason for disagreement is that each expert studies his own favored formulas and draws conclusions from these formulas. Rarely does he test exactly the same paint that someone else is testing. As a result, there is no bridge between his results and the results of other laboratories, or other tests. Our laboratory has made a practice of always exposing the same basic test paint in every series of house paint exposure tests that it has made in the last fifteen years. The results in

<sup>19</sup> Pratt & Lambert, Inc., Buffalo, N. Y.

any one series can be interpreted in terms of other series through this common denominator. However, we cannot interpret our results in terms of tests made by other laboratories.

Has Committee D-1 ever considered the possibilities of setting up a short series of standard reference paints which would be available in liquid form to all laboratories for exposure, immersion, and physical testing purposes? By including one or more of these reference standards in every industrial test, it would not take long to build up an enormous amount of information about these

paints. Beyond that, every test in which the paints had been included could be compared with other tests containing the paints. We feel that this proposal would help to clear up some of the disagreement that exists today.

Elm's proposal to undertake a statistical study in order to pre-determine durability is scientifically sound and certainly worthy of trial, but few companies or laboratories would have the personnel, time or testing facilities to undertake such a study. The industry needs simple testing procedures.

### Author's Closure

Mr. Wirshing suggests that it is practically impossible "to check each panel in a manner so as to be able to guarantee that each spot on the panel is the same as every other spot on the panel." I believe that a degree of uniformity can be achieved which will be fully adequate for most paint testing purposes by following a scheme similar to the one outlined in my paper. However, if Mr. Wirshing should be proved to be correct then there is only one way open to the paint test engineer—namely, to follow the principles laid down by Mr. Cranmer and to subject a sufficient number of panels to simultaneous testing so as to obtain an adequate basis for a statistical evaluation. The inclusion of a standard alone, no matter how well established and how extended the experiences gained with it, is entirely insufficient to guarantee the

reliability of the test results. One standard does not eliminate errors due to nonuniformity of the substrate surfaces, although it may serve as a check on the exposure conditions.

Mr. Wirshing states that the influence of inadequately coated areas such as panel edges upon the behavior of the paint system under test is of considerable interest to the automotive engineer, and that for this reason they prefer not to give the edges of their test panels additional protection. This method of testing does not guarantee that all edges are left inadequately protected to the same degree, and therefore leaves considerable doubt whether a certain effect observed is the result of edge failure, or of an intrinsically poor paint system. To get full value of such tests, it would be preferable to eliminate this element of uncer-

tainty and to scratch the test area in a carefully standardized manner.

One of the comments made by Mr. Clare is of particular interest. He states that a thoroughly sanded and polished steel panel presenting the ultimate in clean steel surfaces wets very poorly with water and gives pronounced break. This observation illustrates rather strikingly one of the points that I tried to make in my paper, namely, that the water-break test does not furnish any information on the cleanliness of the substrate surface, but merely indicates whether it is uniformly hydrophilic in nature. Surface chemical experiments conducted in our own laboratories have shown that a thoroughly clean steel surface is organophilic but becomes increasingly hydrophilic as rusting proceeds.

It is very difficult to fit the results of the water absorption experiment reported by Mr. Wray into the picture which we derived from our own experiments. However, without knowing the exact experimental details of Mr. Wray's experiment, I should like to attempt an explanation. It would be my guess that water-soluble material of some sort was leached from the film specimen immersed in the distilled water and depressed its vapor pressure sufficiently to cause the slightly lower water absorption as compared with the absorption from the vapor.

The bad experiences Messrs. Lutz and Lewis report with waxed edges are probably due to excessively high

temperature of the molten wax used for coating the panel edges. When wax is kept only a few degrees above its melting point and the panel is slightly below this temperature when it is dipped into the wax, creepage of the wax is kept at a minimum. Embrittlement of the wax edges at lower than room temperatures, though rather serious with some waxes, gives no trouble when a good grade of certain wax is used. For tests at very low temperatures the scheme of coating the panel edges with extra coats of paint is preferable.

Mr. Singleton's observations regarding the effect of sanding on the corrosion tendency of steel surfaces as measured by a modified Mears test would be of greater value if he also reported the time elapsed between sanding and testing and the conditions of storage during this interval. It would then be possible to form some idea of the density and nature of the oxide film that very likely formed on the panel surface. In our experiments we have endeavored to avoid such secondary effects as that reported by Mr. Singleton because they tend to becloud the issue. We prefer to hold the panel on a magnetic chuck during the sanding operation since the massive chuck aids considerably in dissipating heat generated. Mr. Singleton's report illustrates strikingly one of the many variables that can be introduced, and should be eliminated or gotten under control if interpretable results are to be guaranteed.



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# A Message from Your Officers

*This Month's Contributor*

**TOM L. HOLCOMBE**

Director, Representing Associate Membership

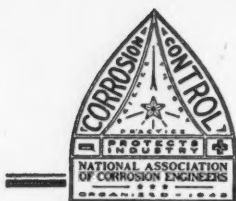
**E**DWIN JEWELL, of Monsanto Chemical Company, and NACE member, lost his life at Texas City. A short time before the fateful and untimely disaster, I had the pleasure of meeting Edwin in Chicago during our last conference. I shall not forget that day because he took the time and trouble to single me out, and for a purpose. In a serious, sober sort of way, Edwin said, "I have some constructive criticism to offer."

Among other things he said, "There are many underlying causes which contribute to corrosion problems, thus we have already many members who badly need help but as yet have not found so much as a lead. Before real accomplishments are attained each member and the industry he represents should be duly considered. Let us not criticize the men who organized NACE nor the officers at the present time but help them concentrate on broadening our scope to where each and all shall benefit. Sure I know more money will be required. I think I have an excellent prospect for you. Every piece of metal in use today is insured—think of the assistance the numerous insurance companies could give us as members and the help we can give them as an association with ways and means to control corrosion. Actually our field is unlimited."

I feel certain it would interest Edwin Jewell today if he knew we now have over 1000 active memberships plus associate and corporate memberships, which exceed 100 in number. I am equally certain it would interest him to know that local sections are being successfully organized, in many instances, without financial aid from the parent organization. That our aims and intentions are good may be well evidenced by our progress, however, the ultimate goal of this association lies a long way ahead. Think how much nearer to that goal we would be if before next conference we doubled all memberships. A few minutes each day from every member undoubtedly could more than accomplish this end.

We are still a young organization, we are not infallible and certainly we are not immune to constructive criticism. Edwin Jewell had the courage of his convictions, he expressed a sincere willingness to work and he was big enough to support the team all the way. While his passing is a distinct loss, I am sure his sound criticism regarding 100 per cent cooperation, with fairness and justice to all, shall not go unheeded.





## NACE News

### MARINE CORROSION PROBLEMS THEME OF HOUSTON SECTION MEETING

The Houston (Texas) Section of the South Central Regional Division of the National Association of Corrosion Engineers met September 9. Approximately 60 members and guests attended the session, the theme of which was Marine Corrosion Problems. Following dinner, a brief business meeting, presided over by Section Chairman W. P. Noser, Humble Oil & Refining Company, was held, during the course of which Donald Bond of the Texas Pipe Line Company was elected Secretary-Treasurer to succeed Glenn King.

The principal speaker of the evening was J. L. Baughman of the Texas Game, Fish and Oyster Commission, who held his audience with an interesting paper titled, "Marine Fouling Organisms." Mr. Baughman described in detail the characteristics and habits of various organisms that inhabit the coastal waters of the United States, citing several case histories and giving detailed information related to their attack upon submerged structures. Branche-Krachy Company, who resigned due to pressure of business.

It was developed during the course of the discussion that followed the reading of the paper, that most of these same organisms have a definite affinity for the paints and coatings used to protect metals under water. These organisms attached themselves to a submerged coated surface, consume the coating and leave the surface exposed. Convincing evidence of this fact was brought out by Walter Rogers of the Gulf Oil Company in his talk on "Corrosion Experiences in Louisiana Brackish Waters." Mr. Rogers stated that corrosion of under water portions of barges operated by his company has proved a serious problem. In all cases it has been necessary to remove all floating equipment from the water at least once every six months to scrape and re-paint the surface. He said that tests run on commercially available marine paints have failed to reveal a one with a life expectancy greater than six months. Corrosion above the water line is of little consequence as compared to that below the water surface, said Mr. Rogers.

L. G. Sarpe of the Humble Oil and Refining Company presented a brief paper on "Marine Anti-Fouling Paints," and R. A. Brannon, also of the Humble Company, described the International Nickel Company's corrosion test site at Kure Beach, N. C. Lt. Commander J. C. Gadberry, U. S. Marine Corps, and assigned to the Texas Group of the Atlantic Fleet, gave a resume of

corrosion prevention methods being used by the Navy, in maintaining the "Mothball Fleet," especially that at the Orange, Texas, anchorage where he is stationed. Commander Gadberry said that the Navy's principal problem in regard to the Mothball Fleet was interior corrosion, which he said was best combated by deaeration.

### GENERAL INTEREST

**Tom L. Holcombe**, Dearborn Chemical Company, Shreveport, La., has accepted the chairmanship of the Committee on Investigation of Protective Coatings, newly established NACE technical research committee. Mr. Holcombe's committee will function under the direction of the Technical Practices Committee, of which R. B. Mears, Carnegie-Illinois Steel Corporation, Pittsburgh, Pa., is chairman.

**George A. Sands**, Electro-Metallurgical Co., unit of Union Carbide and Carbon Co., New York, has accepted appointment as co-chairman of the Chemical Industry Symposium, one of ten technical sessions scheduled during the April 5-8, 1948,

NACE Conference and Exhibition, to be held at the Hotel Jefferson, St. Louis, Mo. M. G. Fontana, Chairman of the Technical Program Committee for the Conference, in making the announcement, stated that Mr. Sands will serve as co-chairman with N. E. Berry under the chairmanship of W. C. Fernelius.

**Battelle Memorial Institute**, Columbus, Ohio, has named six new assistant directors—all veteran members of the Battelle staff. The new assistant directors are Frank C. Croxton, Clarence H. Lorig, Howard W. Russell, Ralph A. Sherman, Clarence E. Sims and John D. Sullivan. These men will share with Director Clyde Williams and Oscar E. Har-

#### *Arrange Schedule for 1948 Conference*

**T**WO AND one-half days of the four-day 1948 Conference and Exhibition sponsored by the National Association of Corrosion Engineers will be devoted to technical sessions. The Conference will open Monday, April 5, with registration in the morning at the Hotel Jefferson. The exhibition arena, located on the mezzanine floor of the hotel, will open officially at the same time, Tuesday, April 6, concurrent technical sessions will be held in the morning, starting at 9:00. And Wednesday and Thursday, April 7 and 8, two concurrent sessions will be held in the morning, starting at 9:00, and two in the afternoon, starting at 2:00. The annual NACE banquet will be held in the Gold Room of the hotel at 7:30 Wednesday evening.

der, assistant director since 1930, in the technical direction of Battelle research. The creation of the new posts amounts to a streamlining of the Battelle organization. Under the Battelle plan, administrative matters will be handled by the director and four assistants to the director. The assistant directors are the top coordinators of technology.

In addition, a body of technical advisors, headed by Horace W. Gillett, chief technical advisor, provides specialized consultation and guidance on all research projects. During the period beginning with World War II, the Institute's staff has tripled in size, growing from 300, to its present 1000. Research expenditures this year are expected to exceed \$4 million.

Upon completion of the second in a series of lectures inaugurated by the Humble Oil & Refining Co., Houston, Texas, for the benefit of its technical and research personnel at Baytown (Texas) Refinery, the company announced that the series of lectures would be resumed in February, 1948, when A. P. Colburn, assistant to the president and advisor on research at the University of Delaware, will conduct a two-weeks course on "Special Topics in

Distillation." Mr. Colburn will be followed later in the spring by Richard T. Arnold, chemistry department of the University of Minnesota, who will discuss "Special Topics in Organic Chemistry." Joel H. Hildebrand, University of California, will appear on Humble's program during the latter half of June, 1948, to lecture on "Thermodynamics." The program will be closed out with two two-week courses in August and September, 1948, by F. A. Matsen, University of Texas, lecturing on "Physical Chemistry." The 1947-48 program got under way August 18 of this year with a two-weeks course conducted by E. R. Gilliland, Massachusetts Institute of Technology, who enlarged on the lecture he gave last year on "Transference of Processes from Small to Large." He was followed by H. A. Laitinen, who conducted a three-weeks course which opened September 2 on "Physical Problems of Analytical Chemistry."

**Bjorksten** Research Laboratories have leased from Edwal Laboratories, Inc., laboratory space at 732 S. Federal Street, Chicago, Ill., in order to provide additional capacity for research projects. The Edwal Laboratories have transferred their research activities related to production problems to their Ringwood, Ill., plant. They are continuing the balance of their research work and customer service laboratories at the downtown Chicago address.

**Centro Research Laboratories**, Briarcliff Manor, New York, announce details of an association with CTAB (Chemisch-Technisch Adviesbureau) of Haarlem, Holland.

### Notice

• Effective January 1, 1948, subscription to the National Association of Corrosion Engineers' journal, **CORROSION**, will be \$7.50 per year, with a \$3.50 per year rate extended to educational and public libraries. The present method of allocating \$3.00 of Members' \$7.50 dues for a subscription to **CORROSION** will continue in force.



CTAB is jointly directed by J. Rinse, one of the continent's leading research workers in the field of organic coatings, plastics, pigments and associated materials; and W. Dorst, a specialist in plant layout, especially design and practical application of paints and other coatings. This combination had developed the processes for, and have built or remodelled numerous paint and varnish plants, and the largest resin plant in Holland. CTAB is credited with extensive studies in the chemistry of both raw materials and intermediates, such as the polymerization, dehydration, vinylation and blowing of linseed, soy bean, tung, oilseed and castor oils, resin modification, chlorination of rubber, the formulation of fungicides and preservatives, and the products of

**T**HE NEWS SECTION was primarily incorporated in *Corrosion* to provide a record of the current activities of members of the Association, and to convey information of interest and value to members. All members are invited (in fact urged) to send releases, or letters, informing the editors of changes in positions, promotions, achievements, or other news items. All material should be forwarded to the Editor of *CORROSION*, 905 Southern Standard Bldg., 711 Main Street, Houston 2, Texas.

pigments and other raw materials. Among the new processes and products Centro Research is interested in are metal putties, plastic coatings for concrete, grease-resisting and shrinkage-controlled textiles, and air drying urea resins.

## PERSONALS

**C. H. McRaven** has resigned from the Dow Chemical Company, Freeport, Texas, to accept a position with the United States Navy in connection with corrosion control of their Panama Canal pipelines. While in the employ of Dow, Mr. McRaven was a research and development engineer and was engaged in the development and application of magnesium for cathodic protection of pipelines. He is the author of several papers on the subject of cathodic protection.

**Christy J. Vander Valk** has been named technical director of the Red Lead Division of the Lead Industries Association, New York, N. Y. Mr. Vander Valk was formerly associated with the Research Depart-

ment of the Heyden Chemical Corporation, Garfield, N. J. His duties in his new position will include coordination of the extensive research program of the Association on metal protective paints and technical service to users, and specifiers of red lead.

**Fred L. Goldsby** has been appointed manager of the Foreign Erection Department of the Chicago Bridge and Iron Company, and transferred from Chicago to the New York office of the company. Mr. Goldsby is a Director of NACE, representing the Corporate Membership, and also Chairman of the General Program Committee for the Fourth Annual Conference and Exhibition, which the Association will stage in St. Louis next April.

**F. M. Ruffing**, Member of NACE, formerly with the Cathodic Protection Sales Division of The Dow Chemical Company, Midland, Mich., announces the organization of the Magnode Products Company, Inc., 139-151 Banker Street, Brooklyn, New York, to manufacture magnesium anodes for the mitigation of corrosion in all types of hot water storage tanks.

**George L. Ericson**, chemist, has been appointed to the staff of the Battelle Memorial Institute, Columbus, Ohio, where he will be engaged in research on industrial corrosion problems. Mr. Ericson received a B.S. in chemistry from Bethany College in 1936. Prior and subsequent to his service as a lieutenant (j.g.) in the Naval Reserve, he was a chemist for the Atchison, Topeka and Santa Fe Railway Company.

**Carlos F. Hickethier** has been appointed head of the new Chemical and Physical Research Department of the Obras Sanitarias de la Nacion of Buenos Aires, Rep. Argentina. Mr. Hickethier only recently returned to Buenos Aires after an extended visit to the United States. In his new position he is charged with forming and directing a special section for corrosion research.

**Richard Pomeroy**, consulting engineer associated with James M. Montgomery, has moved his offices and laboratory to 40 West Green Street, Pasadena, California.

**Duncan C. Pell** has joined the Glaspray Process Co., Inc., San Francisco, Calif., as a research engineer. He was formerly associated with the Plasticoat Company in the same city.

## OBITUARIES

**David A. Russell**, chief chemist for The Youngstown Sheet & Tube Company, Youngstown, Ohio, died at his Youngstown home July 26. A member of NACE since March 1945, Mr. Russell had been engaged in the study of corrosion problems for almost 40 years, and since receiving his B.S. in chemistry from Penn State College.

**Olin K. Smith**, chemical engineer of the United Light and Power Company, died at his Davenport, Iowa, home June 14, following an illness of several months. Mr. Smith, who had been a member of NACE since August, 1944, attended both Pyatt Tech and Columbia Uni-

versity, and had been engaged in corrosion work for more than 26 years.

**Charles F. Meyerherm**, president and chief engineer of Albert F. Ganz, Inc., consulting engineers, died July 27 in the Mount Vernon, N. Y., Hospital, after a brief illness, at the age of 58. He had specialized in electrolysis studies since graduating from Stevens Institute of Technology with a degree in mechanical engineering. A member of NACE since April, 1944, he was also a member of several other technical societies. He is survived by his widow, and a son, Charles Mueller, Jr., of Tuckahoe, N. Y.

## NEW PRODUCTS, EQUIPMENT AND SERVICES

**Announcement** of the development of a new type of corrosion-resistant pipe and fittings was made by the Plastics Division of The Dow Chemical Company. The company will fabricate the new pipe at its Midland plant.

The product, Saran-lined steel pipe, designed to convey corrosive liquids, combines the desirable features of steel pipe—rigidity and pressure strength—with Saran's extreme resistance to chemicals.

Main use of the pipe is in manufacturing chemicals, and in plants such as pulp, paper and textile; metal treating and metal plating.

Saran-lined steel pipe is resistant to a very wide range of chemicals, oils and solvents. It will satisfactorily carry corrosive sulphuric, hydrochloric, nitric and phosphoric acids. The working temperature of the pipe, important to chemical engineers, ranges from 0° to 190° F. depending upon service conditions.

Pipe in maximum lengths of 10 feet and in sizes from 1 to 4 inches will be manufactured. Fittings, also Saran-lined, include elbows and tees, companion and reducing flanges and gaskets. The latter are made of specially compounded Saran to assure maximum chemical resistance at joints.

Field assembly presents no undue problems. Conventional power tools may be used for cutting and threading and assembly of joints is very similar to that of standard steel piping, the company reports.

**Bee Chemical Company**, 63 East Lake Street, Chicago, has developed a corrosion inhibitor, Steelyfe 11, for steel and other ferrous metals.

This product has as its active ingredient the barium salt of an alicyclic sulfonic acid. Not only does the product act as an outstandingly effective corrosion preventive but there is also additional protection against corrosive sulfate ions which react with the barium liberating alicyclic sulfonic acid which in itself is an effective corrosion preventive, according to the company. The new inhibitor, a water displacing corrosion preventive for indoor storage, is a thin, dark liquid having a flash point about 105° F. It can be applied by dipping, brushing or spraying. Upon evaporation, it leaves a transparent coating and thus permits inspecting and measuring. This coating has high corrosion prevention properties and can be readily removed by wiping or with degreasers even after standing for several years. For most indoor protection jobs, one part of Steelyfe 11 is added to six to 12 parts of Stoddard Solvent. The product can be furnished in dilution ready for use.

**Fairchild Camera and Instrument Corporation** has announced the new Fairchild Pneumatic Temperature Measuring System, for measurement and control of extremely high gas temperatures to 5000 degrees, Reaumur. The instrument is a form of gas thermometer utilizing the density of the gas being measured as a direct indication of the temperature. The Fairchild corporation claims this method can solve specific gas measurement-control problems encountered in a broad variety of fields, including aircraft, marine, railway, and general industrial. In-

struments are also available for laboratory use. Specified accuracies for these instruments are plus-or-minus 1 percent to 2500 degrees R., and plus-or-minus 2 percent from 2500 to 5000 degrees R., with a response rate of one-half second or less. Further information can be obtained by writing the Engine Control Division of Fairchild Camera and Instrument Corporation, 88-06 Van Wyck Blvd., Jamaica 1, N. Y.

**Fully self-contained** and very compact, an exhaust waste heat boiler of an entirely new design has been developed by American Locomotive Company, according to Hugh Corrough, director of the Company's Alco Products Division. Enclosed in an insulated steel cabinet only six feet, six inches long; seven feet, six inches high, and three feet, two inches wide, the unit functions automatically on exhaust

gases from a turbocharged diesel engine, or it can be operated by household type oil burner. The waste heat boiler generates 500 pounds of steam per hour on the diesel exhaust and considerably in excess of that figure when operating on the oil burner. The units are manufactured at the Company's plant in Dunkirk, N. Y.

Seventeen of the new boilers are in service at pumping stations along the Great Lakes Pipeline Company route, which stretches from Barnsdall, Oklahoma, via Kansas City to Grand Forks, N. D.

A wide range of application for the new waste heat boiler was pictured by Mr. Corrough, who asserted that not only product pipelines and central power stations but all industries using diesel engines can apply the unit for heating on limited amounts of process steam.

### CORRECT COMMITTEE CHAIRMEN

In the article, "Duties of New Technical Committees," which appeared on page 4 of the September Corrosion News Section, the names of the chairmen of the new committees were transposed. Mars G. Fontana is chairman of the Committee on Corrosion Problems Involved in Processing and Handling Chemicals, while A. V. Smith heads the Committee on Minimum Current Requirements for Cathodic Protection.

## The Necessity of Closer Cooperation Between the Engineer and the Operating Personnel\*

By R. P. Gonzales\*

It has been my good fortune to have attended the National Association of Corrosion Engineers' conventions both at Kansas City in 1946, and in Chicago last April. I went to those meetings with my eyes and ears open, anxious to learn more about this work the members of NACE have cut out for themselves, and learn how best it could be applied to, or help me in my work of building, operating and maintaining a cross-country gas pipeline, and how the operating personnel can help in the problem.

Through attendance at those meetings, I learned that there are a great many people, in fact, thousands—devoting their whole working time to the problem of combating corrosion.

The reason there are a greater number of people devoting their time to this work today is because there is a greater necessity for corporations having as their major capital assets metal structures buried in or resting on the ground, to have on their payrolls and attached to their engineering staffs, men who are well versed in corrosion problems, men willing to devote their time and energy toward protecting these valuable assets. Valuable as-

sets? Yes! Metal structures are much more valuable today than ever before. This increased valuation can be attributed to four present-day conditions. First, high cost of labor and material for replacement purposes, as well as new construction. Second, it is a known fact that the life of a great majority of the existing structures can be economically extended by use of proper cathodic protection, thereby lessening the hazard of interrupted service to customers. Of course, less interruptions mean less maintenance cost and greater revenue. Third, the tendency of oil and gas engineers today (principally gas engineers) is to construct and operate new pipelines at a safety factor as low as 1.3 to 1., and to use metals for tubing having a yield of approximately 55,000 psi and tensile strength of approximately 80,000 psi. This means, of course, that thin wall pipe is used wherever practical, thereby reducing the construction cost, which is the first cost. To maintain these new lines through a long span of life it is essential that thought and plans be given to cathodically protecting the lines before construction begins, or soon thereafter. Fourth, high tax trends; operating and maintenance costs affect the net income of corporations. Management is giving as much thought today, if not more, than it did ten years ago, toward maintain-

\*A paper presented at the May 28, 1947, meeting of the Shreveport (Louisiana) Section of the South Central Regional Division of NACE.

\*Assistant Superintendent, Gas Pipe Line Department, Arkansas-Louisiana Gas Company.

ing a reasonable corporate income. And only by constantly fighting to maintain an income, can a company hope to adequately serve its customers and satisfy the investing public, and only by doing so can it hope to secure new capital for expansion.

Before the corrosion engineer came into being, the few records (and there were very few kept) that were kept of oil and gas leaks and hot spots were those prepared by the operating and civil engineering personnel. This was usually done by indicating on inventory or alignment maps the location, station number, and date on which blind saddles were installed on pipelines to shut off leaks. Hot spots were determined after numerous leaks developed within a short distance on the pipeline. After hot spots developed, plans were then made to replace the section. This was the old method. Today hot spots can be located, by use of scientific instruments, long before it becomes necessary to replace the section of pipe. Soil corrosion and electrolysis can definitely and economically be arrested by cathodically protecting the section.

Where it once was the responsibility of the operating personnel to maintain records of leaks and hot spots, it is now the duty of both the operating personnel and the corrosion engineer. The two departments now make a team; and without harmonious team work, accurate records cannot be secured. It is the responsibility of the operating department to report accurately to the engineer when leaks are found, the date, time, material spent in repairing the leak and the general terrain

in which the leak developed. It is the responsibility of the operating department to make frequent inspection tours of all cathodic units, reporting the day on which the inspection was made, the time spent, miles travelled, and cost of repairs to the units, if any.

It is the duty of the engineer to make field or line surveys before new structures are buried in the soil, and also on old structures as soon after leaks are reported as practical. It is the engineer's duty to thoroughly test the soil potential, to select, with the aid of the operating personnel, sites for ground beds, and to select the type and size of cathodic units best suited for the job. I believe the job of selecting pipe coating and pipe wrapping should be left to the planning engineer, who will no doubt consult with the corrosion engineer, operating personnel and others having wider knowledge of the over-all problems at hand.

It is also the duty of the corrosion engineer to post all information sent in to him and that which he gathers himself to his records. To assemble these data in such a way that a brief, intelligent cost-report of each unit or line can be presented to management. After all, it is the management who must determine the economy of cathodic protection. It is management who must provide in the budget the cost of continuing or enlarging the program. So let us operate on the basis that the corrosion engineer and operating and maintenance personnel make a team, working for the good of the corporation, its customers and its investing public.





# Corrosion Abstracts

## ATMOSPHERIC CORROSION

**Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals.** ASTM Spring Meet. Pittsburgh, Jan. 27, 1946.

A symposium held under the sponsorship of ASTM Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys to evaluate and correlate the data resulting from the extensive atmospheric exposure tests on metals and alloys carried out by Comm. B-3 for 10 years. The following papers by various authors are included: The Corrosion of Rolled Zinc in the Outdoor Atmosphere; The Behavior of Nickel and Monel in Outdoor Atmosphere; Resistance of Copper Alloys to Atmospheric Corrosion; The Use of Lead and Tin Outdoors; The Resistance of Aluminum-Base Alloys to Atmospheric Exposure; and Tracking Troubles in Atmospheric Corrosion Testing. Also included are the 1943 and 1944 reports of ASTM Subcomm. VI on Atmospheric Corrosion of Non-ferrous Metals and Alloys.

**Determination and Effect of Sulfur Gases in Plant Atmospheres.** B. J. Sweo and M. J. Bozsini, Ferro Enamel Corp. Paper before Am. Ceramic Soc. 49th Annual Meeting, Chicago, April, 1946; Am. Ceramic Soc. Bull., **26**, 3, 69 (1947) Mar. 15.

Methods for the determination of sulfur gases in plant atmospheres were devised and analytical data were obtained. The chemical analysis of enamel at various stages of processing indicated that unfired cover coat absorbed sulfur gases and that the degree of absorption was proportional to the time of exposure. This observation was corroborated by simulative test data. The primary sources of sulfur gases in the plant studied were: 1) burning furnace; 2) driers; 3) pickle room, and 4) compressed-air supply. As a secondary source, the liberation of absorbed sulfur gases from the ware during firing is indicated. The effect of these gases on scumming of cover-coat enamel is discussed.—INCO.

## CHEMICAL CORROSION

**Corrosion.** MARS G. FONTANA. Ind. & Eng. Chem., **39**, 91A-92A (1947) Mar.

Discusses the prevention of corrosion caused by fluorine and by hydrofluoric acid.—BLR.

**Corrosion on Gasworks.** A. J. BRANDRAM. Gas World, **126**, 369-74 (1947) Mar. 29.

Several types of differential corrosion may occur on gasholders. On gasholder crowns, corrosion occurs most frequently at the point where the radial joint between two sheets

is overlapped by a sheet of the next inner ring. Corrosion four to six inches below the water level is also noted on the water seals of gas-holders. The use of the ferroxyl test to determine anodic and cathodic areas is suggested. The importance of relative humidity and industrial pollution is pointed out. It is suggested that gasholder corrosion may be greater on the sunny side. Under marine conditions of atmospheric exposure, mild steel appears to be considerably more resistant to corrosion than any of the wrought irons, and the very pure charcoal iron is the most corrodible. When exposed to industrial atmospheres, British wrought irons are more resistant to corrosion than mild steel. Swedish iron (and ingot iron) is still the most corrodible. Copper-bearing steel has been suggested for gas-holder plates, with a view to minimizing corrosion, but has been used to only a limited extent. Metal surface preparation is discussed, with descaling methods. The weathering method followed by wire brushing gives a very poor foundation for paint. Metal spraying with zinc or aluminum is of great value. Sufficiently thick coats require no paint protection, but such paint protection is generally desirable. Red lead is a very satisfactory pigment for metal priming; red oxide of iron paint should include red lead or chromate primer or be used only for finishing coats. The adhesion of chlorinated rubber paints has been improved. Bituminous paints are good for waterproofing, but deteriorate in sunlight. Coal tar may be used, but it should be dehydrated and about 10 percent lime added. It is applied hot, or cut back with a

solvent. The discussion brought out other instances of corrosion.—CEC.

**Corrosion.** MARS G. FONTANA. *Ind. & Eng. Chem.* **39**, 83A-84A (1947) Feb.

Discusses economics of materials for chemical plant construction and desirability of standardizing means of expressing corrosion resistance of materials. Recommends adoption of "mils/yr." by all concerned.—BLR.

**Aids to the Selection of Corrosion Resisting Piping Material.** L. G. VANDE BOGART. *Valve World*, **43**, 5, 130-135 (1946) Nov.-Dec.

The author gives and discusses a suitable questionnaire to assist in the selection of piping material. Attention is also drawn to the author's "Guide to the Selection of Corrosion-Resistant Piping Materials" published in the same journal, May, 1944.—BNF.

**Discussion of Paper on Chemical Corrosion Resistance of Lead.** H. H. UHLIG. *Corrosion*, **3**, 149-150 (1947) Mar.

Points out that lead or lead alloys should not be used in equipment coming in contact with food and beverages because of the toxicity of lead-corrosion products. The author states that this point was not mentioned in "Chemical Corrosion Resistance of Lead" published in the December, 1946 issue.—BLR.

**Estimating Chemical Piping Costs.** R. J. SCHRADER. *Chem. Eng.*, **24**, 109-110 (1947) Jan.

Tables give estimated prices for metal, porcelain, glass, and plastic (Saran) fittings and tubing. Formula for estimating installation costs (based on required man-hours) is presented.—BLR.

# ~~Corrosion~~ BARRED



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# *Reilly* Protective Coatings

## COATINGS

**Interim Descriptive Statement on the Leaching-Rate Test for Ship's Anti-Fouling Compositions.** Iron & Steel Inst., Joint Tech. Panel on the Leaching-Rate Test of the Marine Corr. Sub-Comm., Aug. (1946); Paper 19, (1946), Advance copy, 5 pps.

The test assesses the value of anti-fouling compositions by measuring the rate of loss of toxic ingredients from the paint surface during immersion in sea-water. Originally developed for application to anti-fouling compositions containing cuprous oxide as the only toxic ingredient, the test is being adapted for use on formulations containing more than one toxic constituent and in particular on those with copper and mercury. Test procedure is described.—INCO.

**Moulds and Bacteria in Paint, From the Point of View of a Microbiologist.** H. J. BUNKER. J. Oil & Colour Chem. Assoc. **29**, 201-214 (1946) Sept.

Reviews factors governing the development of micro-organisms; the causative organisms; coatings and the anaerobic corrosion of metals; anti-fouling compositions; and properties of paint anti-septics. 16 ref.—BLR.

**Electrochemistry of Protective Films on Metals. Investigation of the Behavior of Aluminium as a Cathode.** E. N. PALEOLOG and G. V. AKIMOV, *Compt. rend. de l'acad. des Sciences (USSR)* **51**, 609-612, (1946) (Cf. Abst. 105, 1947); *Chem. Absts.* **41**, 6, 1565 (1947).

Aluminium as cathode in alumi-

nium-zinc and aluminium-magnesium couples in a 0.04 normal sodium chloride solution buffered with sodium acetate (pH 5.2) are studied, and current produced is measured. The efficiency of aluminium cathode with air film or pickled in nitrogen sodium hydroxide or anodized 0.15 and 3 thickness was studied. Findings are reported with curves, analysis of which confirms that a large part of the natural protective film on the surface of aluminium is electrochemically inert in solution. The area of electrochemically active spots is less on anodized surface than on aluminium with protective film formed in the air.—ALL.

**New Paint Medium for Use on Light Metals.** Light Metals (England) **10**, 111, 175-176 (1947) Apr.

During the war years, Germany developing a class of organic compounds known as the polyurethanes. It now appears that these materials, when used as paint media, possess an outstandingly good adhesion to light metals. The polyurethanes are resinous materials obtained by reaction between polyisocyanates and various classes of organic compounds containing reactive hydrogen atoms capable of replacement by sodium. The coatings produced by the polyurethane resins can be pigmented in the usual way, the two essential constituents being marketed complete with pigments, solvents and diluents and are mixed immediately before use when reaction begins at once and proceeds rapidly. Films take from five to 24 hours to dry, according to conditions and the actual constituents employed, but they will stove to



## How RED LEAD "PASSIVATES" METAL ...inhibits RUSTING

Research discloses sound reasons for Red Lead's long acceptance as the "standard" metal protective paint.

None is more important than Red Lead's ability to keep iron and steel in a "passive" state, in which the rusting process is held to a minimum.

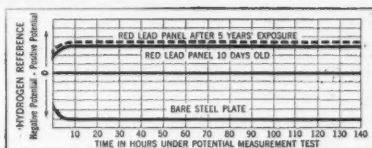
As you know, unpainted steel, exposed to moisture, rapidly rusts. However, the same metal, under identical exposure conditions, remains "passivated," or rust-inhibited, when coated with Red Lead.

The graph at right shows clearly the rust-inhibitive effect of Red Lead on steel, as contrasted with the rapid and continuous rusting of bare metal.

It is worth noting that, even after five years' exposure, the "passivating" power of Red Lead is still retained. Small wonder, then, that maintenance men think of Red Lead *first* when it comes to making metals *last*.

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### RED LEAD'S Protection Measured Electrically

A piece of unpainted steel was immersed in water. The iron, going into solution, reacted with oxygen in the water to form rust. This unrestrained corroding state is indicated by a rapidly developed and maintained negative potential relative to hydrogen (see graph).

But, when steel panels painted with Red Lead were immersed, iron and lead salts formed next to the metal. This action at once stifled corrosion by preventing the iron from going into solution, thus keeping the steel surface passive. Graph curves show a positive potential, quickly rising and constantly maintained.

\* \* \*

**NATIONAL LEAD COMPANY:** New York 6; Buffalo 3; Chicago 8; Cincinnati 3; Cleveland 13; St. Louis 1; San Francisco 10; Boston 6 (National Lead Co. of Mass.); Philadelphia 7 (John T. Lewis & Bros. Co.); Pittsburgh 30 (National Lead Co. of Pa.); Charleston 25, W. Va. (Evans Lead Division).



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**RED LEAD**

hard elastic finishes in  $\frac{1}{2}$ -1 hour at 160-200° C. (320-392° F.) In addition to their hardness, elasticity and good adhesion, the coatings are characterized by their excellent durability, resistance to water, chemicals and organic solvents, non-inflammability, and excellent electrical characteristics, that is to say, high specific resistance and breakdown voltage.—ALL.

**The Pigment-Binder Relationship as a Fundamental Property of Paints.** A. W. F. THYNNE. *Paint Technol.*, **11**, 423-427 (1946) Nov.

Reviews oil absorption techniques; relationship of absorption to particle size and shape; oil absorption of pigment mixtures; the packing system of pigment; wetting of pigment by oil; effect of acid value; and metal protective paints. 28 ref.

—BLR.

**Porosity of Electrodeposited Metals.** A. E. S. Research Project No. 6. Part I. Bibliography. N. THON and E. T. ADDISON, JR. *Monthly Rev.*, **34**, 445-453 (1947) Apr.

Includes only references which bear directly on the porosity aspect in corrosion. Bibliography is arranged chronologically and, for each year, in the alphabetical order of the names of the authors.—BLR.

**Use of Metallic Pigments in the Preparation of Protective Paints.** J. E. O. MAYNE. *J. Soc. Chem. Ind.*, **66**, 3, 93-95 (1947) Mar.

The use of paints containing metallic pigments for the protection of iron against corrosion is considered in this paper. A study has been made of the conditions which must

be met in order for metallic pigment to protect iron cathodically. These conditions are: 1) The metallic pigment must have an electrode potential less noble than that of iron, and 2) The pigment particles must be in metallic contact with each other and with the iron. It has been shown that while aluminium powder, zinc dust, and magnesium powder satisfy the first condition, only zinc dust meets the second condition.—CALCO.

**Protection Against Corrosion by Means of Chromium Diffusion Zones.** G. BECKER, K. DAEVES and F. STEINBERG. *Iron & Steel Inst. Translation No. 296*, 7 pp. From *Z.V. d. I.*, **85**, 127-129 (1941) Feb. 1.

Describes the chromising ("Inkromierung") process for rendering a surface layer of steel stainless by treatment with a solid, liquid or gaseous chromium-bearing substance; the active agent is usually chromous chloride. A "case" of high chromium steel, 0.1 mm. deep, can be produced in a few hours.—BNF.

**Metallic Diffusion into Iron in the Solid State from Sprayed Coatings.** P. BARDENHEUER and R. MÜLLER. Translation from *Mitt. K.-W. Inst. Eisenforschung*, **14**, 295-305 (1932).

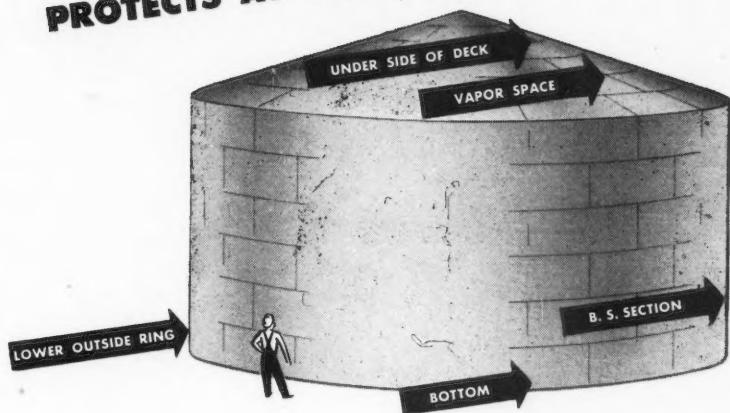
Investigation of diffusion from sprayed coatings of nickel and chromium, together and separately and with or without aluminum and silicon. Nickel diffused readily; aluminum, though diffusing itself, prevented diffusion of other elements. Role of vapour phase in diffusion is studied.—BNF.



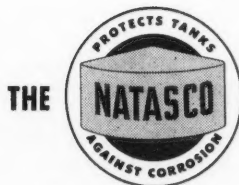
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## CONSTRUCTION MATERIAL

**Investigation of Electrolytic Corrosion of Steel in Concrete.** Corrosion, **3**, 37-54 (1947) Jan.

Progress Report of Committee on Electrolysis of the Association of American Railroads, include studies of corrosion and current flow in several railroad foundation pedestals and the establishment of a suitable test procedure for determining resistance to ground, amount of current flow, and corrosion of steel embedded in concrete. Results are described and shown in photographs and tables. Several important conclusions are drawn. Appendix describes a study of the effect of electric current on concrete.—BLR.

**New Strong, Nonmagnetic Spring Material has High Corrosion Resistance.** Matl. & Methods, **25**, 4, 94-95 (1947) Apr.

Elgiloy, a new watch spring material developed by Elgin with Battelle Memorial Institute and other companies, is reportedly composed of: 20% chromium, 40% cobalt, 15.5% nickel, 15% iron, 7% molybdenum, 2% manganese, 0.15% carbon. It is remarkably ductile, offers excellent workability either hot or cold, is nonmagnetic, unaffected by finger prints, high humidity and shows no rust after 500 hours, exposure to 10% salt spray, as well as surviving hot akua regia for 12 minutes without harmful effect. Although now restricted to watch springs because of limited output, apparent possibilities include razor blades, stainless cutlery, surgical and dental instruments, gun springs, valve parts, chemical equipment, automotive valve springs and parts,

and electronic components. The armed services are said to be investigating its use in jet propulsion and in gas and steam turbine equipment.—INCO.

**Graphic Heat Exchangers. Part I.** C. E. FORD. Chem. Eng., **54**, 92-96 (1947) Jan.

Corrosion resistance of graphite to a large number of chemicals is outlined.—BLR.

**Failures of Railway Materials by Fatigue.** H. O'NEILL. Symposium on the Failure of Metals by Fatigue: Univ. of Melbourne, (1946) Dec.

The types of fatigue failure met with in rails, types, wheel-centres, axles, fire-boxes and stay-bolts are discussed and illustrated. The accelerating effect of corrosion and the influence of non-metallic inclusions are considered. Fatigue failure of parts in transit is instanced.—BNF.

**An Engineer Discusses Merits of Ceramic Glazed Clay Pipe.** H. W. JEWELL. Brick & Clay Rec., **110**, 59-60 + (1947) Mar.

Some sizes of unglazed clay pipe were found to exceed ASTM specifications by 50 percent. Ceramic glazings of bore added strength.

—BLR.

**Sulfide Sulfate Corrosion of Cements.** A. M. KUZNETSOV. J. App. Chem. (USSR) **19**, 5 and 6, 489-491 (1946) (In Russian.)

The simultaneous action of hydrogen sulfide and sulfates on cements is investigated. Results indicate that aluminum cements of similar chemical and mineralogical composition are the most resistant.

—BLR.



## ***Curb Corrosion*** **... by using TRANSITE PIPE** **for salt-water disposal lines**

**O**IL men know Transite as the pipe that curbs corrosion... outside, inside and all the way through! It has proved remarkably resistant to the corrosive action of salt water on the inside, corrosive soil on the outside. That's why you'll find this asbestocement pipe so widely used for salt-water disposal lines.

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without mechanical handling equipment. Its factory-made Simplex Couplings speed assembly, provide joints that *stay* tight in service. Flexible, they permit laying the pipe around curves without special fittings.

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# **Johns-Manville** **TRANSITE PRESSURE PIPE**

## GAS AND CONDENSATE WELL CORROSION

**Results of Some Studies of the Condensate Well Corrosion Problem.** WALTER F. ROGERS and HARRY E. WALDRIP, Gulf Oil Co. Corrosion, **3**, 121-140 (1947) Mar.

Results of a study of the wetting power of condensates and waters for steel in the presence of each other, with and without wetting agents. Resulting effect on the corrosion rate of the mixture was studied by laboratory methods and the testing was extended to field operations.

—BLR.

**Summary of Research on Drill Stem Performance.** THOMAS J. YOUNG, Drilling Contractor, **3**, 47-49 (1947) Feb. 15.

The five reports of the Battelle Memorial Institute to the Association of Oilwell Drilling Contractors, are condensed in this summary. Summary affords a general background for the widening forthcoming Battelle activity for the Association during 1947.—BLR.

**Progress Report on Drill String Research.** L. R. JACKSON, H. M. BANTA, and R. C. McMASTER, Drilling Contractor, **2**, 43-52 (1946) May 15; Ibid 52-65 (1946) Aug. 15; Ibid 46-60 (1946) Nov. 15.

Three separate reports from Battelle Memorial Institute to the American Association of Oilwell Drilling Contractors present: first, results of a metallurgical examination of specimens of pipe from the Permian Basin which had failed on account of corrosion; second, a survey of methods and a discussion of problems involved in non-destructive

testing of drill pipe, including recommended procedure for improving the quality of mill and field inspection; and third, results of extensive laboratory tests of methods for improving the operating life of drill string under corrosive-brine conditions. The three most promising methods were found to be: metallic plating, plastic coating, and addition of chemical inhibitors.—BLR.

**New Technique Combats Electrolysis.** R. B. WALTER, Oil Weekly, **124**, 18-21 (1947) Feb. 3.

A 15-foot rod on which was strung an alternating series of copper and zinc plates, separated by brass spacers, is installed in corrosive wells in a position to make intimate contact with the oil being pumped. The casing and pump parts are protected against corrosion by the cell couples set up between the copper and the zinc. The "treater units" must be replaced occasionally when the zinc plates are used up.—BLR.

**Corrosion in High-Pressure, Gas-Condensate Wells.** H. A. CARLSON, Phillips Pet. Co. Oil & Gas J. **45**, 33, 81-4 (1946) Dec. 21.

A review of the literature on gas-condensate well corrosion problems is presented in an attempt to correlate and summarize the data found therein. The review is divided into sections covering the following: acidity of well water, effect of temperature and pressure, velocity and turbulence, metal-ion concentration, nature and concentration of dissolved substances, electrostatic discharges in the fluid, physical and chemical nature of the metal-protective films, prevention and control of corrosion (suitable corrosion-resist-

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*Method*  
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Corrosion engineers agree that cathodic protection is the best all-round method of keeping corrosion rates at the lowest possible levels. And General Electric Rectifiers are just about the finest equipment to do the job.

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\*According to the  
National Association of Corrosion Engineers,  
as reported in *Business Week*, April 19, 1947.

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ant alloys, inhibitors), corrosion-testing and detection, calculations of corrosion rates, and suggestions for investigation and research. The Humble procedure for the electro-metric titration of fatty acids in water is included. 10 ref.

**Chemical Treatment of Distillate Wells—Practical Aspects.** T. S. BACON, Lone Star Producing Co., Dallas, Texas. *Corrosion*, **2**, 4, 188-193 (1946) Oct.

Type of inhibitor, injection methods, evaluation of effects are discussed, on the basis of the experience of the Lone Star Producing Co.—INCO.

### GENERAL CORROSION

**Corrosion Control.** W. H. J. VERNON. Paper before London Section, Soc. of Chem. Ind. Iron & Coal Trades Rev., **153**, 677-678 (1946) Oct. 18; *Chem. & Ind.*, 1946, 389 Oct. 26; *Corr. & Mat. Prot.* **4**, 2, 26-27 (1947) Mar.-Apr.

Describes recent work, since 1934, of the Chemical research laboratory of the Dept. of Scientific and Industrial Research, not hitherto published. Many problems, often of interest to Service Departments, have been investigated, e.g., corrosion by rapidly flowing sea-water and protection of ships' bottoms; corrosion in circulatory systems, in enclosed spaces and by anti-freeze solutions and the development of inhibitors; accelerated tests for atmospheric corrosion; microbiological corrosion. The high-speed rotor technique for investigating the effect of movement on rate of corrosion is able to discriminate rapidly between good and bad compositions of ship's-bottom paints. Investigation of the cathodic



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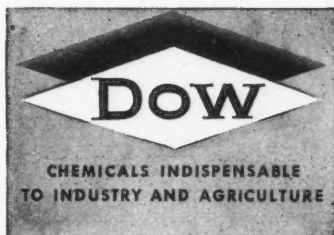
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protection of steel in sea-water by means of zinc showed that weight of steel saved was more than three times weight of zinc lost. Sodium benzoate in anti-freeze concentrations is a fairly efficient anti-frigerant and is virtually non-corrosive toward metals used in radiator systems and is also effective for protecting metals from corrosion by wrapping materials and is a fungicide. Anaerobic corrosion of ferrous metals in soils, resulting in graphitization of cast-iron, opened up a new field of investigation, presenting quite different problems from those of immersed or atmospheric corrosion. 17 ref.

**Metal Conservation—A National Problem.** C. GERHARD DAVIDSON. *Metals*, 17, 11-14 (1947) Jan.

Country's natural reserves of anti-

mony equivalent to 4 years' supply; lead, 12 years; cadmium, 16 years; zinc, 19 years; copper, 34 years; platinum, 2 years and tungsten, 4 years.—BLR.

#### METAL FAILURE

**Corrosion Resistance of 27 Percent Chrome Alloy Recorded High in Plant Service Tests.** *Pet. Proc.*, 2, 116-17 (1947) Feb.

A series of tests conducted for the Office of Rubber Reserve of Babcock & Wilcox, and several oil companies including Phillips Pet., Shell Oil, Union Oil, Standard Oil (Calif.) and Sun Oil companies on the use of 27% chromium-iron alloy in butadiene manufacture are reviewed. The formation of sigma phase or "885° F. embrittlement" or both resulting in brittle failures of steel

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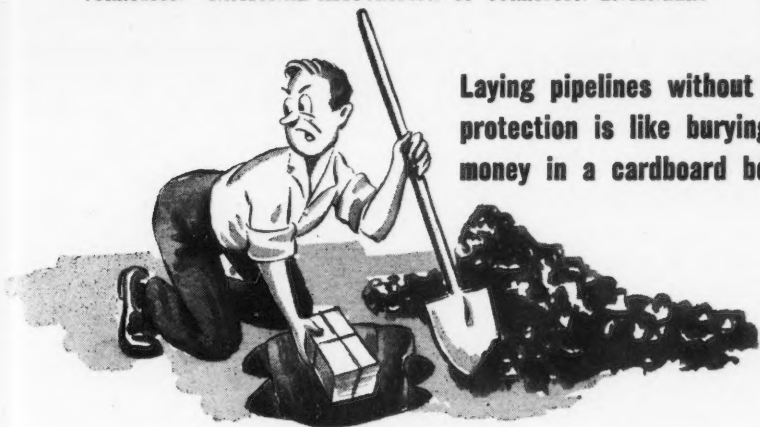
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pipng in such processes must be avoided by a number of precautions including preheating before welding, avoidance of sharp notches or re-entrant angles and shock stresses in fabrication and repair, rapid cooling through the 1100-700° F. range in service and prevention of overheating in catalyst regenerating cycles. Heat treatment at 1500° F. or higher restored ductility in sheets and plates of this alloy by dissolving the sigma phase. Similar investigations of silicon and nickel-chrome steels are considered briefly. This alloy was studied for use in catalytic dehydrogration processes.

**Corrosion Causes Most Cylinder Wear. Digest of "Minute Amounts of Cylinder Wear Are Measured with a Microscope,"** by CLARENCE S. BRUCE and JESSE T. DUCK. SAE Journal, **55**, 62 (1947) Apr.

Discusses conclusions and test setup.

**The Corrosion of Iron and Steel and Its Prevention.** J. C. HUDSON. J. Oil Colour Chem. Assoc. **30**, 320, 35-52 (1947).

A general survey of the corrosion of iron and steel and of methods for its prevention is presented here. This survey is largely based on the experimental work of the Corrosion Committee of the Iron and Steel Institute and the British Iron and Steel Research Association. Particular attention is devoted to the subject of atmospheric corrosion. The survey is divided into the following sections: 1) The corrosion of bare iron and steel; 2) general discussion of protective measures against rusting; 3) general discussion of protective coatings for iron and steel; 4)

protective coatings for heavy structural iron and steelwork; and 5) protective coatings for light gauge iron and steel parts.—CALCO.

## PIPE CORROSION

**A Magnetic Thickness Gage.** A. SCHURINGA. *Metalen*, **1**, 1, 10-11 (1946) Sept. 15. (In Dutch.)

For determination of thickness of non-magnetic coatings on steel even in positions difficult of access.

—BNF.

**Application of Forced Drainage Attenuation Constant.** M. E. PARKER, JR. *Corrosion*, **3**, 151-154 (1947) Apr.

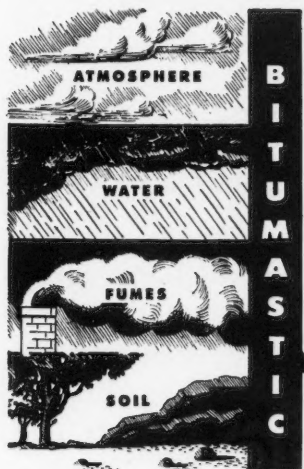
In article by Robert Pope, in the December issue, a set of equations was derived expressing the attenuation of the three quantities: change in structure/soil potential; current on structure; and cathodic current-density; as functions of the distance X along the structure from the drainage point. This paper describes the application of the equation to pipeline with an asphalt-enamel coating. Discussion by Mr. Pope.

—BLR.

**Protecting Pipe in Open Systems.** M. D. APPLEMAN, Asst. Chief Soil Biology, Univ. of Ill. Am. Gas Assoc. Monthly **29**, 4, 194-6 (1947) Apr.

A review of the literature is given relative to pipe corrosion, especially bacterial and means of minimizing. In some cases cellulose wraps have been attacked by bacteria and have led to accelerated corrosion of pipes and cables. Much more needs to be known about anaerobic bacterial corrosion. On a theoretical basis, it is suggested that backfills containing lime are apt to encourage the growth

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of these bacteria. The use of chemical sterilizing or inhibiting agents to limit the growth of these bacteria is suggested—much work needs to be done in this connection, and the use of "pit experiments" is suggested, with heavy inoculation with the sulphate reducing bacteria, and inspection once a year. Methods of compacting the soil around the pipe as a possible means of limiting corrosion need to be studied. (It is noted that a patent has been applied for the use of bactericidal and bacteriostatic agents in soil stabilization by H. F. Winterkorn.) Bibliography.—CEC.

## SURFACE PREPARATION

**Mechanism of Metal Cleaning.** S. SPRING AND L. F. PEALE. *Met. Prog.* **51**, 1, 102-106 (1947) Jan.

It is claimed that observation of the break-up of oil-films on metals in the cleaning process with alkaline cleaners indicates that saponification

and emulsification are not (as has been thought) primary phenomena in the cleaning process. Physical explanations of the cleaning process are put forward, which it is claimed lead to considerable savings in the use of cleaning solutions. Gives results of a study of the mechanism of metal cleaning. Direct visual and photographic observations were made of the process of oil removal from metal.

**A New Express Method of Phosphating Steel Parts.** G. V. AKIMOV AND A. A. ULYANOV. *Comptes Rendus (Doklady de l'Academie des Sciences de l'U.R.S.S.,* **49**, 7, 497-499 (1945); *Bull. Iron & Steel Inst.*, **129**, 76 A (1946) Sept.

A brief account is given of a method of improving phosphate coatings on steel by producing a more uniform, thin, and porous film, and subsequently impregnating it with a chromate solution and solutions of organic substances.—INCO.

*Abbreviations at end of abstracts indicate contributors cooperating in conjunction with the American Coordinating Committee on Corrosion, and the National Association of Corrosion Engineers to reproduce all abstracts pertaining to corrosion under one cover, in the NACE journal, Corrosion, and are as follows:*

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